

The Journal *of the* SOCIETY OF DYERS AND COLOURISTS

Volume 72 Number 11

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THE SOCIETY OF DYERS AND COLOURISTS
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The MSS. of all papers communicated to or read before the Society become the property of the Society. They should be addressed to the Editor, Society of Dyers and Colourists, 19 Piccadilly, Bradford 1. Authors must not allow their papers to be published elsewhere before they have appeared in the Society's *Journal*. Should prior publication take place without the sanction of the Publications Committee, the paper will be printed only as an abstract or summary.

Manuscripts submitted for publication in the *Journal* should be typewritten (double spacing) on good-quality paper, using one side of the paper only and leaving a margin at least 1 in. wide on the left-hand side. The time taken in refereeing papers (both lectures and communications) will be reduced to a minimum when authors submit two copies of the typescript.

In view of the high costs of publication, it is essential that authors should be as concise as possible. When experimental procedure has already been published, a literature reference to the paper containing the details is sufficient, whilst well known experimental methods should be described very briefly.

Introductory paragraphs describing the aims of the investigation and the method of attack are desirable, and should be followed by the experimental results and their discussion. There should be a brief summary for insertion at the beginning of the paper. References to the literature should be numbered consecutively, using superscript numbers without brackets immediately following the text words or author's name to which they refer.

The list of references should be given at the end of the manuscript and the abbreviations used should be, as far as possible, those given in the "List of Periodicals Abstracted" included at the end of the index to the preceding year's *Journal*. Reference numbers in this list should be neither enclosed in brackets nor followed by full-stops. As far as possible throughout the manuscript the abbreviations listed in the Jan. 1950 issue (p. 54) should be used. Tables should be numbered consecutively in Roman numerals and figures in Arabic numerals.

The number of figures and graphs should be kept as low as possible, and data should be presented in the form of either tables or graphs, not both. Drawings should be carefully prepared, preferably in Indian ink, on plain white drawing paper or, preferably, Bristol board. In graphs, the frame and actual curves should be ruled and inked more heavily than any co-ordinate lines, and the latter should not be close together as in ordinary graph paper. Experimental points should always be given, and where several graphs appear in a single figure clear means of differentiation must be adopted. All numbers and legends are set up in type by the printer, and authors should therefore indicate them lightly in pencil.

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Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1-6 of the January 1956 and pages 313-317 of the July 1956 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (Telephone Bradford 25138-9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURE

Some Early Stages in the Renaissance of
the British Dyemaking Industry

C. M. Whittaker

COMMUNICATIONS

In the Footsteps of Perkin

W. H. Cliffe

Light Fastness Assessments of Dyed Textiles
and their Bearing upon the Mechanism of Fading

J. C. Eaton and C. H. Giles

Separation of Disperse Dyes by Paper Chromatography

K. Elliott and L. A. Telesz

Absorption of Alkali by Wool Keratin from Aqueous
Solutions of Sodium Carbonate and Bicarbonate

R. P. Harker

JOURNALS WANTED

The Society is urgently wanting Journals for all months of 1955 except July and December. The months of August and September 1955 are particularly required. Copies of January, February, March, and April 1956 issues are also in demand. Please address communications to the
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FORTHCOMING MEETINGS OF THE SOCIETY

Tuesday, 27th November 1956

LEEDS JUNIOR BRANCH. *Some Investigations into the Application of Dyes to Acrylic Fibres.* B. Kramrisch, Esq., F.R.I.C., F.T.I., F.S.D.C. (Clayton Dyestuffs Co. Ltd.). Departmental Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, Leeds 2. 3.30 p.m.

Thursday, 29th November 1956

BRADFORD JUNIOR BRANCH. *Petroleum Chemicals in the Textile Industry.* M. Bratherton, Esq. (Shell Petroleum Co. Ltd.). Technical College, Bradford. 7.15 p.m.

Tuesday, 4th December 1956

MANCHESTER JUNIOR BRANCH. *Developments in Analytical Methods in the Dyehouse Laboratory.* R. W. Richardson, Esq., B.Sc., Ph.D., A.R.I.C. and Z. Bergman, Esq. (Courtaulds Ltd., Coventry Dyehouse). Manchester College of Science and Technology, Manchester. 7 p.m.

Thursday, 6th December 1956

WEST RIDING SECTION. *Reactive Dyes—A New Approach to Cellulose Dyeing.* T. Vickerstaff, Esq., M.Sc., Ph.D., A.R.I.C., F.S.D.C. (I.C.I. Ltd., Dyestuffs Division). Victoria Hotel, Bradford. 7.30 p.m.

Friday, 7th December 1956

LONDON SECTION. *The Chemical Revolution in Textile Technology.* Professor J. B. Speakman, D.Sc., F.R.I.C., F.T.I. (Leeds University). (Joint meeting with the London Section of the Textile Institute.) Royal Society, Burlington House, London, W.1. 6 p.m.

Tuesday, 11th December 1956

HUDDERSFIELD SECTION. *Problems of a Commission Dyer.* H. V. Partridge, Esq., M.Sc., A.T.I. Silvios Cafe, Westgate, Huddersfield. 7.30 p.m.

SCOTTISH SECTION. *Full Shades on Secondary Acetate and 'Tricel' and Allied Topics.* H. C. Olpin, Esq., M.Sc., F.R.I.C., F.S.D.C. and J. Wood, Esq., B.Sc., A.R.I.C. (British Celanese Limited). St. Enoch Hotel, Glasgow. 7.15 p.m.

Wednesday, 12th December 1956

MIDLANDS SECTION. *The Finishing of Garments and Fabrics of Bulked Terylene.* G. McLeavy, Esq., B.Sc., Ph.D., A.R.I.C. King's Head Hotel, Loughborough. 7 p.m.

NORTHERN IRELAND SECTION. *Printing of Nylon and other Polyamide Fibres.* R. E. Fletcher, Esq., B.Sc., A.T.I. (The Clayton Dyestuffs Co. Ltd.). Thompson's Restaurant, Donegall Place, Belfast. 7.30 p.m.

Friday, 14th December 1956

MANCHESTER SECTION. *Pigment Printing—The Possibilities and Limitations.* Dr. Kass (Fabriken Bayer, A.G.). Textile Institute, 10 Blackfriars Street, Manchester. 7 p.m.

BRADFORD JUNIOR BRANCH. Lecture. Details later.

Wednesday, 9th January 1957

NORTHERN IRELAND SECTION. *Application of Colour Physics to Textiles.* P. H. Oliver, Esq., B.Sc., Grad.Inst.P. and J. C. Guthrie, Esq., M.Sc., F.Inst.P. (Courtaulds Ltd.). Thompson's Restaurant, Donegall Place, Belfast. 7.30 p.m.

Thursday, 10th January 1957

MIDLANDS SECTION. *Developments in Analytical Methods in the Dyehouse Laboratory.* R. W. Richardson, Esq., Ph.D., B.Sc., A.R.I.C. and Z. Bergmann, Esq. (Joint with the Society of Chemical Industry). Gas Theatre, Nottingham. 7 p.m.

Thursday, 10th January 1957

WEST RIDING SECTION. *Full Shades on Secondary Acetate and "Tricel" and Allied Topics.* H. C. Olpin, Esq., M.Sc., F.R.I.C., F.S.D.C., J. Wood, Esq., B.Sc., A.R.I.C. (British Celanese Ltd.). Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Friday, 11th January 1957

LONDON SECTION. *Ladies' Evening. Kippers, Cocktails, Confectionery & Colour.* G. J. Chamberlin, Esq. (The Tintometer Ltd.). Royal Society, Burlington House, London, W.1. 6 p.m.

Monday, 14th January 1957

BRADFORD JUNIOR BRANCH. *The Use of Fading Lamps for Determining Light Fastness.* P. R. Dawson, Esq. (I.C.I. Ltd.). Technical College, Bradford. 7.15 p.m.

Tuesday, 15th January 1957

SCOTTISH SECTION. *Application of Colour Physics to Textiles.* J. C. Guthrie, Esq., M.Sc., F.Inst.P., F.T.I. and P. H. Oliver, Esq., B.Sc., Grad.Inst.P. (Courtaulds Ltd.). St. Enoch Hotel, Glasgow. (Joint Meeting with Textile Institute.) 7.15 p.m.

LEEDS JUNIOR BRANCH. *Structural Aspects of Cellulosic Fibres.* Dr. N. S. Wooding (Courtaulds Ltd.). Departmental Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, Leeds 2. 3.30 p.m.

Friday, 18th January 1957

LONDON SECTION. *Annual Dinner and Dance.* Waldorf Hotel, London W.C.2. 7 for 7.30 p.m.

MANCHESTER SECTION. *A New Approach to the Dyeing of Hydrophobic Fibres.* D. Garrett, Esq. (I.C.I. Ltd.). Textile Institute, 10 Blackfriars Street, Manchester. 7 p.m.

WEST RIDING SECTION. *Ladies' Evening.* Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Tuesday, 22nd January 1957

HUDDERSFIELD SECTION. *Application of Colour Physics to Textiles.* J. C. Guthrie, Esq., M.Sc., F.Inst.P., F.T.I. and P. H. Oliver, Esq., B.Sc., Grad.Inst.P. Silvios Cafe, Westgate, Huddersfield. 7.30 p.m.

Wednesday, 23rd January 1957

MIDLANDS SECTION. *Problems in the Processing of Rayon Blends.* R. C. Cheetham, Esq., A.M.C.T., F.R.I.C., and H. D. Edwards, Esq., Ph.D., A.R.I.C. College of Technology, Leicester. 7 p.m.

Tuesday, 29th January 1957

BRADFORD JUNIOR BRANCH. *The Work of a Colourist in a Man-made Fibres Dyehouse.* J. T. Lynes, Esq. (Courtaulds Ltd.). Technical College, Bradford. 7.15 p.m.

Thursday, 31st January 1957

WEST RIDING SECTION. *Discussion, Detergency.* Panel—R. S. Hartley, Esq., A.R.I.C. (Wool Industries Research Association), G. H. Rostron, Esq., A.R.I.C. (Joseph Crosfield & Sons Ltd.), R. C. Tarring, Esq., B.Sc., A.R.C.S. (Shell Chemicals Ltd.). Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Friday, 1st February 1957

LONDON SECTION. *A Review of the Colour Fastness Requirements in Dyed Textiles.* T. H. Morton, Esq., M.Sc., Ph.D., F.S.D.C. (Courtaulds Ltd.). (Joint meeting with the London Section of the Clothing Institute.) Royal Society, Burlington House, London W.1. 6 p.m.

* Tuesday, 5th February 1957

LEEDS JUNIOR BRANCH. *Supply, Treatment and Disposal of Water in Relation to the Dyehouse.* R. W. Richardson, Esq., B.Sc., Ph.D., A.R.I.C. (Courtaulds Ltd.). Departmental Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, Leeds 2. 3.30 p.m.

continued on page xxxix



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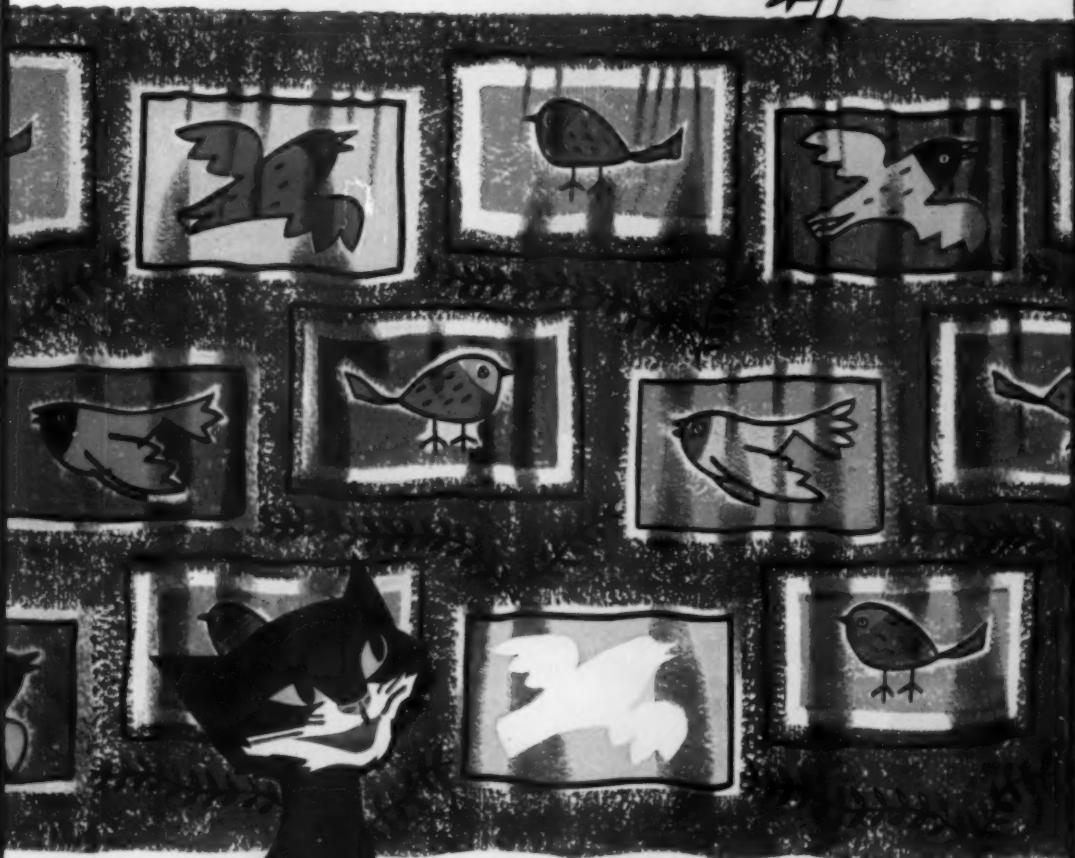
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THE JOURNAL OF THE Society of Dyers and Colourists

Volume 72 Number 11

NOVEMBER 1956

Issued Monthly

Proceedings of the Society

THE THIRTEENTH JOHN MERCER LECTURE

The Disperse Dyes—Their Development and Application

R. K. FOURNESS

*Meeting held at the St. Enoch Hotel, Glasgow, on 13th April 1956,
Mr. Fred Smith (President of the Society) in the chair*

The disperse dyes have come to be applied to a very large number and variety of fibres and have therefore achieved an importance which is probably greater than the weights used would suggest. They were the first type to be applied directly to a fibre from an insoluble suspension. Their preparation includes a process of particle size reduction and often subsequent drying, both in the presence of a dispersing agent. The mechanism of dyeing with them would seem to entail their passing from suspension through solution on to the fibre, to which they become attached by non-polar forces.

The colouring matters used in the preparation of disperse dyes belong mainly to three chemical groups. The connection between dyeing and fastness properties on the one hand and chemical constitution on the other is in general indefinite. However, the best fastnesses are obtained by moving in the direction of greater molecular complexity, but this unfortunately is accompanied by a reduction in dyeing power. The introduction in recent years of the man-made fibres has accelerated the development of new application methods, and these are assisting the dyer to utilise these more complex dyes. This tendency emphasises that the achievement of better fastness with disperse dyes will require, as with other dyeing classes, a close understanding between, and the combined efforts of, both those working on the elaboration of new dye structures and those whose interests lie in one or other aspects of dye application.

INTRODUCTION

The connection between the subject of this lecture and the man whose name it commemorates is at first sight, perhaps, difficult to see. John Mercer was 64 years old, an age when most men either have already retired from active work or are about to do so, before the synthetic dye industry was born with Perkin's great discovery. The disperse dyes, one of the youngest of the large progeny which resulted from that discovery, did not come until sixty years later, while the idea of making fibres instead of growing them did not find practical expression until Chardonnet's patent nearly twenty years after Mercer's death.

Where, then, lies the connection? It could be maintained that the discovery by Ellis and by Baddiley and Shepherdson of the "disperse" technique would not have been possible without Mercer's invention¹ in 1846 of the sulphonated oils. But the causal relationship of such events is often a reality only in the mind of the historian. Can it be doubted that some lesser chemist might not have stumbled on this one of Mercer's many discoveries if the "Father of Textile Chemistry" himself had overlooked it; or that Holland Ellis might not have sought out some other agent to accomplish his purpose?

Such surmise is unnecessary. The story of the disperse dyes, like the rest of dye and dyeing technology, abounds in examples of invention and discovery which were the results of just that combination of acute perception and inventive genius possessed in overflowing measure by Mercer. This is the link for which we are looking—a link which has throughout the ages joined all those possessed by the restless urge, to invent and to improve.

DEFINITION AND EARLY HISTORY

The Society has recommended² that the term *disperse dyes* shall mean: "a class of water-insoluble dyes originally introduced for dyeing cellulose acetate, and usually applied from fine aqueous suspensions". The definition is gratefully accepted by the author, for taking the term in its wider meaning would have meant expanding this lecture to an unreasonable length. It would have had to include much else—the modern forms of the vat dyes, for example, and perhaps even many of the organic pigments used in printing inks, paints, and plastics. The limitation will permit a more coherent story to be told of a group of dyes without which certain man-made prodigies would have been stillborn or at best remained Peter Pans.

The disperse dyes were not the first colouring matters to be applied from a suspension. Alizarin and certain of the so called Alizarin dyes were so used, but they were applied to materials previously mordanted with metallic salts. The disperse dyes were the first type to be applied from a virtually insoluble suspension *directly* to a fibre, and thus represented a break with the principle that dyes must be soluble or capable of being made soluble before dyeing. The Ionamines³, which were the immediate precursors of the disperse dyes, were of this latter type, being in effect "solubilised disperse dyes". Solubility in water was achieved by converting the primary or secondary amino groups of insoluble dyes to *N*-methanesulphonic acid groups. Under the conditions of dyeing these hydrolysed to reproduce the original dye, which was then taken up by cellulose acetate. The device was not entirely satisfactory in practice. The rate of hydrolysis varied from dye to dye, for example, and made the application of mixtures troublesome.

From the experience gained with the Ionamines and with the few dyes from the existing ranges which up to then had been used for cellulose acetate came theories relating chemical constitution and affinity for this new fibre. It was considered that suitable dyes should be (a) "basic" in character and (b) have a relatively simple chemical structure. Such prerequisites were found to imply low solubility in water—it seems obvious in the light of present-day knowledge that this first example of a hydrophobic fibre would require dyes of a like character—and the next part of the task was to achieve some means of "solubilisation" that would not interfere with affinity and other properties.

In 1923 the problem was solved by the chemists of four British firms. Within months of one another they patented methods based on a principle which has since been widely adopted for many and diverse purposes outside the dye industry, e.g. for the preparation of insecticides and water paints. Holland Ellis of British Celanese Ltd. devised a means of producing fine aqueous suspensions of a group of water-insoluble azo and diphenylamine dyes and demonstrated how simply and directly they could be applied to cellulose acetate. He used "sulpho fatty acids", e.g. sulphoricinoleic acid, or their alkali-metal salts⁴, and thus were born the S.R.A. dyes, named after the agent used to disperse them. Baddiley and Shepherdson of British Dyestuffs Corporation Ltd. (B.D.C.) dispersed a series of aminoanthraquinone compounds by grinding or dissolving them in a solvent and precipitating in the presence of soap or Turkey Red oil⁵. These were the original Duranols. A. J. Hall⁶ found that cellulose acetate could be dyed in aqueous suspensions of nitrodiphenylamines containing e.g. Turkey Red oil or soap. The Celatenes of Scottish Dyes Ltd., which were later discontinued, were also aminoanthraquinones and hydroxyanthraquinones⁷.

Ellis's method, consisting in dissolving the dye in an oil or a sulphonated oil, pouring into aqueous alkali, and sieving into the dyebath, entailed no

more preparation than is expected of the dyer who applies vat or azoic dyes. Dyemakers, nevertheless, true to the tradition Perkin left behind, sought and found ways of preparing these dyes in a form that eliminated this complication and gave the dyer a product as simple to prepare and to apply as the direct cotton dyes. So successful was the "disperse" technique that many other dyemakers entered the field, using various dispersing agents, until today many ranges are available to the dyer. These include the Acetoquinones (Fran), the Amacels (Celanese Corp. of America), the Artisils (S), the Celanthrenes (DuP), the Cellitons (BASF), the Cibacets (Ciba), the Dispersols and the Duranols (ICI), the Eastmans and the Eastones (Eastman Kodak), the Serisols (YDC), the Setacyls (Gy), the S.R.A.s (BrC), and the Supracets (LBH).

The growth in the importance of the disperse dyes is illustrated by the increase in their use in the U.S.A., where production rose from 1.2 million lb. in 1934 to 8.4 million lb., or 4.5% of the total production of all dyes, in 1951⁸. A similar story could no doubt be told of the United Kingdom and other textile-manufacturing countries.

THE DISPERSION PROCESS

In the last stage of their synthesis most of the coloured compounds used as disperse dyes separate as relatively large particles, often of a crystalline character. If used in this form they would be quite unsuitable for dyeing, producing uneven and perhaps "specky" results. They would not, moreover, yield their full tinctorial power. This has been demonstrated by Vickerstaff⁹, who found that the quantity of 1-anilino-4-methylaminoanthraquinone absorbed by cellulose acetate in 24 hr. is increased one hundredfold by grinding the crystalline dye in a ball-mill for 48 hr. Bird and Manchester¹⁰ also have emphasised the importance of small particle size in obtaining maximum colour yield from dyes which do not build up well. That a similar relationship holds in printing is also the conclusion of Daruwalla and Turner¹¹. Thus, in order to obtain in practice maximum colour yield, reproducibility, and freedom from faults, the dye must be presented to the fibre as a fine, uniform, stable suspension. This means not only reducing all dye particles present to less than a certain size, but also having present a dispersing agent, which will effectively maintain these particles in individual suspension until they are taken up by the fibre, and which will itself not be affected by temperature, hardness of water, or presence of dyeing assistants.

A simple method of reducing particle size is to dissolve the dye in e.g. sulphoricinoleic acid as used by Ellis, or a solvent such as ethanol, and then to pour the solution into water containing a suitable surface-active agent. The use of solvents is not commercially practicable—the solvent is expensive besides being troublesome to deal with, and many dyes are not sufficiently soluble. Various methods are used by different dyemakers, but they all have one feature in common, viz. the necessity to use quite large amounts of mechanical energy to bring about the necessary reduction in particle size.

One method used in Germany¹² consisted in first mixing the dye filter-cake with a concentrated solution of the dispersing agent. The resulting paste was next partly dried on a drum dryer, and the product thus obtained was remoistened to give a sticky, dough-like mass. This was fed between rollers set close together and revolving at different speeds. The shearing action so produced broke down the larger dye particles to the required size. Further drying and grinding to a powder completed the process.

Another method is to rotate, in a large porcelain-lined cylinder or mill containing flint pebbles or balls of porcelain or steatite, a slurry of dye filter-cake containing dispersing agent. Rotation continues until microscopic examination of a sample indicates that almost all the dye is in the form of particles not larger than 4μ in diameter, which requires 10–60 hr. With dyes of a highly crystalline character, either this milling process is too slow or it fails to produce a dispersion from which the full tinctorial power can be obtained. Recourse must then be had to some treatment of the dye before milling. A useful device is the one known as "acid-pasting", i.e. dissolving in concentrated sulphuric acid, pouring the solution into water, and filtering off the precipitated dye. This, of course, is applicable only to those dyes which are not affected by sulphuric acid at ordinary temperatures. Other difficulties arise at times from the rise in temperature of the mill and its contents, due to the friction of the grinding action. Some dyes soften or become plastic, and in this condition cannot be ground. The slight solubility in water of others enables crystal growth to occur during milling. It is necessary in either of these circumstances to use some means of cooling the mill and its contents.

A satisfactory dispersion consists of particles the diameter of which is 4μ or less. Commercial dispersions often contain a small proportion of larger particles, but these should be kept to a minimum both in size and in number, particularly for printing. Good dispersions of suitable dyes are satisfactory for cake dyeing, as might be expected from the success achieved with dispersed vat dyes by Abbot and Cox^{13,14} and by Hampson¹⁵. Though there does not seem for most purposes to be any practical advantage in reducing the upper limit of particle size of a disperse dye below 4μ , there is a growing tendency for manufacturers to do this. An example of the possibilities in this direction is provided by the Indanthren Colloisol (BASF) vat dyes, which are claimed to be made up of particles ranging from 10μ to 100μ . The lower of these limits approaches the order of size of the aggregates present in solutions of direct cotton dyes. It is of interest to note here that similar considerations apply in the coloration of plastics, though the order of size is different. Certain disperse dyes without dispersing agent, for example, are used for colouring polystyrene. Small beads of the colourless plastic are mixed with the dye before injection moulding. If the dye contains particles larger than about 40μ ,¹⁶ colour specks are likely to occur in the resulting article.

Formerly all disperse dyes were marketed in the

form of pastes, because a satisfactory way of producing a powder which would reproduce the original dispersion on wetting back was not known, and some of the dispersing agents used, e.g. sulphorcinoleic acid, will not dry to a satisfactory powder. Except for special purposes such as printing, most of them are now sold in the form of powders. These are prepared by drying the milled paste in the presence of an excess of dispersing agent. A very satisfactory apparatus for doing this is the spray dryer, the principle of which is to spray the paste into a stream of hot air. Removal of the water present is rapid, and the fine powder that results is not "baked" by remaining in contact with the hot gases. Satisfactory as most modern disperse-dye powders are, it should be remembered that they are, nevertheless, preparations containing a coloured compound having only slight solubility in water. Some care in their use is therefore necessary. Certain of them are sensitive to maltreatment in the concentrated conditions of pasting-up and may aggregate if—and some dyers regrettably still do this—they are boiled at this stage.

A dispersing agent is not necessary for the adsorption of a suitable dye by, for example, cellulose acetate. Dyeing will take place if the fibre is immersed in a suspension of the dye in warm water. Such a method would not, however, suit the dyer. The result would not be uniform and much of the dye would be wasted. It would be impossible, moreover, for the dyemaker to supply the dyer with a usable and stable product. The dispersing agent present in commercial disperse dye powders may be regarded as the vehicle in which the dye proper travels from the dispersing plant of the dye factory to the textile fibre being dyed. Its main functions are—(i) to assist the process of reducing the size of the dye particle, (ii) to enable the dye to be prepared in the form of a powder when this is required, (iii) to facilitate the reverse change from powder to dispersion when the dyebath is prepared, and (iv) to maintain the dispersion during dyeing. In the last of these functions, help is given by dyebath assistants such as soap, which the dyer normally uses when applying disperse dyes.

Various types of surface-active agent will perform these functions, though with differing degrees of success. Examples are soap powder¹⁷ and sulphite cellulose lye¹⁸. More widely used today are the condensation products of naphthalenesulphonic acids and formaldehyde, which are powerful dispersing agents and which dry well. A variation of this type is the agent used in the Celliton range, which is prepared by condensing cresol with formaldehyde in the presence of sodium sulphite and then further condensing with 2-naphthol-6-sulphonic acid¹⁹.

THE DYEING PROCESS

It is only in recent years that the process of dyeing with disperse dyes has been studied to any large extent. Early workers carried out experiments in which cellulose acetate was allowed to remain in contact with aqueous solutions of various

simple organic substances, e.g. phenol²⁰ and *o*-nitroaniline²¹. The results obtained by measuring the amounts of these substances taken up by the acetate from solutions of different concentrations were interpreted as meaning that cellulose acetate behaves under these conditions as a water-immiscible solvent. During the frantic search for dyes for cellulose acetate, it was observed that those colouring matters which had a high solubility in organic solvents, particularly acetone and ethyl acetate, also dyed the new fibre. This lent support to the view that such dyes behaved in a similar way. Kartaschoff²² came to the same conclusion following microscopic observation of the dyeing process, and found support²³ in measurement of the distribution of a series of anthraquinone derivatives between cellulose acetate and ethanol. Thus the process of dyeing cellulose acetate with disperse dyes came to be interpreted as one of solid solution.

Burns and Wood²⁴, who measured the uptake by cellulose acetate of three aminoanthraquinones from aqueous dispersions prepared with gelatin, obtained results which suggested that the mechanism was similar to that of dyeing cellulose with direct cotton dyes, viz. one of adsorption from true solution. Later work by Vickerstaff and Waters²⁵ with a different series of anthraquinone derivatives dispersed in a sulphated fatty alcohol solution gave isotherms which at first sight supported this view. Further investigation, however, led to the conclusion that the dye forms a colloidal solution in the acetate. More recently still, results obtained by Wahl, Arnould, and Simon²⁶, Daruwalla and Turner¹¹, and Bird and Manchester¹⁰ favour the solid solution theory again.

This apparent inconsistency no doubt arises from the difficulty of working with dyes which are present in one phase as undissolved particles, since the results obtained must be dependent to some extent on the state of the dispersion and the dispersing agent used. Perhaps it does not really matter which theory is adopted, for, as Vickerstaff states²⁷, "the distinction between solid solution and adsorption is very largely unreal".

What does emerge from all these studies is a picture of the way in which disperse dyes are taken up by the various hydrophobic fibres. It seems that the dye slips, molecule by molecule, through narrow pores in the fibre structure and attaches itself possibly by means of non-polar van der Waals forces²⁸. These occur between the hydrophobic surfaces of dye and fibre and require a close fit of one to the other. The older concept²⁹ of attachment by hydrogen bonding of the dye to the -CO- groups in cellulose acetate and the polyesters, and possibly the -NH- groups in nylon, perhaps tells part of the story too, it being suggested²⁸ that the formation of such bonds eliminates water molecules bound to the fibre at these points, thereby allowing the close approach of dye and fibre surfaces necessary for the non-polar forces to take effect. In the process of dyeing, then, the dye must almost certainly pass into the fibre via solution in water. Bird³⁰ has shown that all disperse dyes possess some solubility in water at the

temperatures used in dyeing, those with the lowest solubility being the slowest dyeing.

The manner in which a disperse dye dyes under a given set of conditions varies from one fibre to another. Thus, though Terylene (ICI), given sufficient time, is able to take up more dye than can nylon and almost as much as secondary cellulose acetate, it does so much more slowly at temperatures up to 100°C. This is another way of saying that the dye diffuses more slowly within the Terylene fibre than within the others. To the practical dyer, the speed with which a dye is taken up is of far more consequence than its saturation limit for the fibre. He therefore judges Terylene to have "poor affinity", and looks around for some method of improving the dyeing (or diffusion) rate.

Apart from dye selection, there are two approaches to the difficulty. One is to make the fibre more permeable or accessible. By its swelling action the water of the dyebath does this with the natural fibres, with viscose rayon, and with nylon. With the cellulose acetates, too, it suffices unless dyes of limited dyeing power are to be applied for some special purpose, when a swelling agent which temporarily enlarges the pores or intermolecular spaces is used. With the more difficult polyester fibres, dyeing assistants, which have been given the name "carriers", are used. As might be expected from the choice of a different term, these do not behave in quite the same way as the swelling agents. Their mode of action is not clearly understood. One suggestion³¹ is that they act as "molecular lubricants", reducing the cross-linking forces in the fibre and so allowing the dye molecule to force its way in.

The second approach to the difficulty of applying disperse dyes to certain of the hydrophobic fibres is to speed the diffusion by increasing the temperature. There are great theoretical possibilities here: thus, providing that dye and fibre are able to withstand it, as much may be achieved in a second at 200°C. as would take a week at 60°C.³² Temperatures of 200–230°C. for several seconds are used in the Thermosol (DuP) or "dry-heat" process, but so far in this country the main effort has been directed towards using lower temperatures than this. Dyeing rates of a similar order to those of secondary acetate at 85°C. are obtained with Terylene at 120–130°C., and this has stimulated great interest in practical methods of achieving this temperature, which involves working under pressures of about 25 lb./sq. in.

An important factor in dyeing with disperse dyes, and one which has only comparatively recently become the subject of fundamental study, is the rôle of the dispersing agent. The dyer, in contrast to his forebears of thirty years ago, is now confronted with an almost bewildering array of surface-active preparations, and is probably not always aware of the influence of the type and the amount of agent used on the course of his dyeing process. Bird *et al.*³³ have shown the value of such studies, and it is clear from their results that, while the percentage exhaustion at equilibrium of all dyes is lowered by the presence of dispersing agent, the factor of importance in practice, viz.

the rate of dyeing, is affected in a manner which depends on the dye and on the nature of the dispersing agent. Thus, where the dye is virtually insoluble in water at the dyeing temperature—i.e. its solubility is less than 0.2 mg./litre—the rate of dyeing is increased in proportion to the increase in aqueous solubility conferred by the dispersing agent. Examples of this type in current commercial ranges are to be found among the faster dyes of relatively low affinity. With dyes of greater solubility—say above 5 mg./litre at 80°C.—the effect of the dispersing agent is to reduce the rate of dyeing in a manner which is analogous to the retarding effect of certain non-ionic agents on leuco vat dyes. Certain "soluble" dyes, however, behave like the "insoluble" ones. This is explained by assuming that these exceptions are taken up by the fibre more rapidly than the dye particles can dissolve in the dye liquor, and hence they dye more rapidly in the presence of a means—the dispersing agent—of facilitating dissolution of the dye.

It is to be expected that similar considerations apply to the dyeing of other hydrophobic fibres with disperse dyes, though at higher temperatures there may be significant differences. Bird and Manchester¹⁰ have already pointed to one such possibility, viz. the decomposition of the dispersing agent.

From what has already been discussed it is clear that, ideally, each dye would best be applied to each fibre in the presence of a definite proportion of a particular dispersing agent determined by careful studies of the type carried out by Bird *et al.* This ideal cannot be realised in practice, for several reasons. Thus dyes are not often used singly, and as marketed contain a dispersing agent chosen by the dyemaker to give a powder of the right dyeing strength which will readily re-form to a satisfactory dispersion. The dyer, again, usually sets his bath with an agent which is different from the one contained in the dye. This does, however, give him limited control over the best choice. In odd cases the dyemaker deprecates the use of certain agents with a given dye; e.g. Eastman Fast Blue GLF should not be applied in the presence of a non-ionic type of agent.

THE BEHAVIOUR OF MIXTURES OF DYES

It seldom happens that the dyer is able to obtain the required shade with a single dye, and so the effect of one disperse dye upon another, if any, deserves attention.

Breakdown of the dispersion due to interaction between disperse dyes is, fortunately, rare. Though different makers use different dispersing agents, these are mostly of the anionic type, and interference is unlikely. There have been odd cases in the past, usually in the short liquors of the jig, where two dyes have formed a mixture melting at a temperature lower than the melting point of either, resulting in the formation of a tar in the dyebath. With the improvement, over the years, of purity and selection, however, most of the compounds now used as dyes have melting points

above 140°C., and this provides an adequate margin of protection against such disastrous consequences.

Of greater importance is the effect which one dye may have on the dyeing properties of another. Many dyes are influenced by the presence of others and in admixture are not taken up to the same extent as when dyed singly. Others, as reported by Vickerstaff³⁴ in a study of experimental dyeings on nylon and cellulose acetate, are not so affected. This latter additive behaviour may be used to advantage to promote the building-up of deep colours. The components of a mixture of two or more structurally related dyes behaving in this way give the impression of assisting one another on to the fibre. Mixtures of the blue anthraquinone derivatives I and II (Fig. 1), dyed under practical conditions on secondary acetate, for example, produce heavier shades than can be obtained with either of the individual dyes. This is illustrated in Fig. 1, which includes also a range of dyeings of the unsymmetrical dye (III), which might be regarded as combining the two dyes I and II *within the molecule*. As can be seen, it has even better building-up characteristics than a mechanical mixture of I and II. Patents³⁵ based on the use of mixtures of this kind have been granted. On the same effect probably rests the success of certain "homogeneous" disperse dyes. During the manufacture of these, related derivatives are formed in addition to the main product, and the final dye is in fact a mixture of them all. A well known and much used "homogeneous" disperse blue, for example, can be separated chromatographically into at least three components, and the colouring matter present can be shown to contain less than 50% of the product having the structure usually attributed to the dye. It was no doubt with this effect in mind that a famous German dye chemist once humorously remarked to the writer: *So macht ein talentloser Chemiker gute Azetatfarbstoffe*.

The range of dyeing rates exhibited by commercial dye dispersions, though not so great as that of the direct cotton dyes, is nevertheless wide enough to give trouble if care is not taken in the selection of the components of a mixture. This is particularly important when dyeing on the jig, where the material to be dyed is presented portion by portion to the dispersion at dyeing temperature. This may be imitated in the laboratory by replacing the pattern in the dyebath by an undyed pattern at intervals of, say, one minute. The resulting series of dyeings gives an exaggerated picture of the changes which take place in the dyeing properties of the jig liquor, and may be used to determine the suitability of a given mixture for application on the jig. Fig. 2 compares two browns of similar shade but differently constituted: one maintains its tone as the dye is taken up by the fibre, i.e. it is a satisfactory mixture for jig dyeing; the other changes in tone, i.e. it is unsuitable for dyeing on the jig. The process of jig dyeing in general, is, incidentally, one which does not appear to have been the subject of that fundamental study to which its complexity and its widespread use entitle it. Also of consequence is the relative

effect of temperature on dyeing rate. Many listing and ending faults in the dyeing of disperse dyes on the jig arise from neglect of these two factors.

Determinations of relative dyeing rates have been made³⁰, and these provide information which assists in the selection of dyes to be used in admixture. Unfortunately, it is not sufficient to choose dyes which have been shown to have the same or similar rates of dyeing (velocity coefficient), since these, for a given fibre and a given temperature, vary with the concentration of dye used. Vickerstaff³⁷ has stated that it may be assumed, as a rough working guide, that the velocity coefficient varies inversely with the initial concentration. Fig. 3 shows a comparison, by the minute-dyeing technique already described, of two greens composed of different proportions of the same yellow and the same blue: the one on the left-hand side is considered to be satisfactory; the lower proportion of yellow used in the one on the right leads to a greater relative rate of dyeing of this component, resulting in a yellow strike. In order to minimise this effect, a more slowly dyeing yellow, if available, would be preferred. As, however, the choice of dyes is restricted, often by shade and fastness requirements, it is frequently not possible to work entirely to these precepts. The development of methods of application in which differences in dyeing rate are of no consequence, and to which reference will be made later, will be of great assistance in such cases. In the meantime many dyemakers try to assist by making available ready prepared mixtures—browns, greys, navy blues, greens, etc.—in which attention has been paid to these various requirements and the best compromise made.

THE DYE MOLECULE

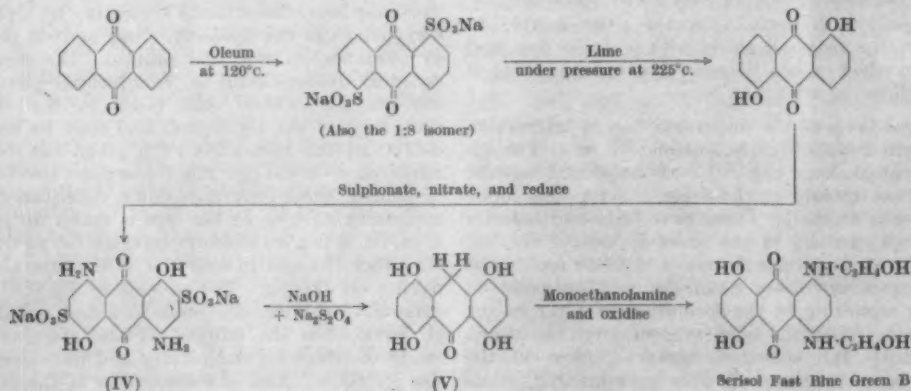
So far we have seen what part the substrate and what part the dispersing agent play in dyeing with disperse dyes. Important as these are, however, it is the chemical character of the dye molecule itself which is the dominant factor in determining the behaviour during the dyeing process and those properties which are concerned with the colour of the resulting material; just as, one might say, the abilities and the disposition of a man are more important than his environment in guiding the course of his career.

The disperse dyes belong for the most part to three chemical groups—(i) the azo group, containing mostly monoazo derivatives with at least two or three disazo dyes, e.g. Dispersol Fast Orange B, and providing a full range of colours including developed blacks; (ii) the nitrodiphenylamine group, yellows and oranges; and (iii) the anthraquinone group, comprising derivatives of α -aminoanthraquinones and providing orange to greenish-blue dyes. Outside these groups are—Duranol Brilliant Yellow 6G (3-methoxybenzanthrone), interesting in that it contains no nitrogen; Celliton Brilliant Yellow FF, obtained by condensing *m*-xylydine with 4-amino-1:8-naphthalic anhydride; a small group of methin dyes prepared by condensing substituted benzaldehydes with cyanoacetic esters, of which only Celliton Fast Yellow 7G (and its equivalents) is important; and two derivatives of 5-amino-8-hydroxy-1:4-naphthaquinoneimine, viz. Celliton Fast Green 3B³⁸, the only homogeneous green, and Artisil Direct Blue GFL³⁹.

Though the disperse dyes as a class are constitutionally simple by comparison with other classes, their manufacture has its own problems. Most require a series of separate chemical operations, diverse in character and requiring plant and equipment of many kinds. As with other types of dyes, faults may arise at any of these stages—for the dyemaker has his D.&K. problems, too—and much patient detective work is then required. Many of the operations require the use of large quantities of acids and solvents, with their attendant corrosion and handling problems.

Serisol Fast Blue Green B affords a good example of a disperse dye the preparation of which is relatively complicated. Starting with anthraquinone, four distinct intermediates are made in sequence before the last stage, in which the dye itself is formed. Following this come the milling and drying operations, which have already been described. Each intermediate requires a number of operations such as reaction, precipitation, and filtration. A summary of the chemistry of the manufacture of Serisol Fast Blue Green B is given below.

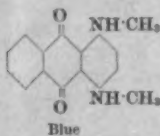
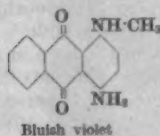
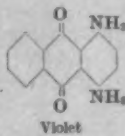
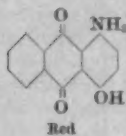
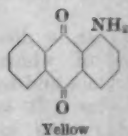
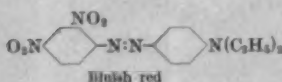
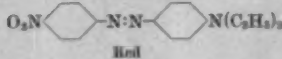
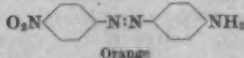
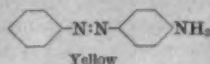
Two points of interest in this series of reactions are—(i) The product IV is the well known acid dye Alizarin Brilliant Blue B (C.I. 1054), which here



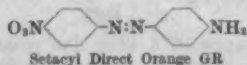
serves as an intermediate: this is the reverse of what is usual with disperse dyes, many of which are themselves intermediates for the preparation of acid, vat, and sulphur dyes. (ii) In the conversion of IV into V (leuco-tetrahydroxyanthraquinone), which is carried out in one short operation, three chemical changes occur, viz. (a) replacement of two NH_2 groups by OH , (b) loss of two SO_3Na groups and their replacement by H , and (c) reduction of the quinone to its leuco form.

CONSTITUTION AND PROPERTIES

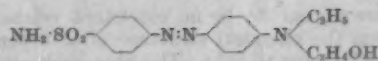
The property of a dye which is first considered is its colour. In the disperse dyes the influence of molecular complexity and substituents on colour follows the general rules observed in other colouring matters. This is exemplified in the following two series of compounds, all of which either are, or could be, dyed as disperse dyes—



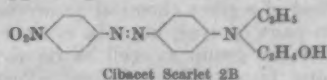
When a dyer speaks of the "colour" of a dye, he means the colour produced by that dye on a given fibre. Hence it is necessary in defining the property of colour to refer to the substrate. This can be a matter of importance with the disperse dyes. Some of them, for example, give different shades on the different fibres to which they are applied. This is particularly true when nylon is compared with either of the acetates: some orange dyes dye redder and duller, and some reds bluer and duller, on nylon. Because of this there was at one time a complete absence of satisfactory bright oranges and scarlets for this fibre. The orange dye for cellulose acetate—



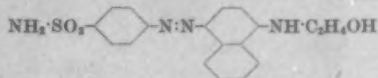
for example, dyes nylon a dull scarlet. By replacing the nitro group with a sulphamoyl group, as in—



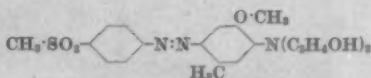
a dye which produces similar bright oranges on the two fibres is obtained⁴⁰. A similar story can be told of the scarlets. The conventional cellulose-acetate types, e.g.—



dye nylon dull crimson. Bright scarlets can, however, be obtained with dyes containing no nitro group, e.g.⁴⁰—



and



Before further discussion of the effect of chemical structure on properties, it will be useful to list the more important properties which are required of a disperse dye. In considering any dye the dyer's first concern is how easy it is to apply. Ideally, dyeing should be rapid, migration (i.e. levelling) good, and the dye capable of producing deep colours, i.e. it should build up well. There should generally be only small differences in dyeing properties within a given range of temperature—say 50–80°C. for cellulose acetate—in order that the temperature variations which occur during dyeing, e.g. on the jig, will not result in unevenness.

The requirements asked of goods dyed or printed with disperse dyes vary, but one or more of the following fastness properties are always called for—light, washing, water, burnt gas fumes, perspiration, sublimation, sea water, chlorinated water, and the organic solvents used in dry cleaning. At times the dye or dyes used must be dischargeable or be resistant to a crease-resist process. Where the dyed material is to be made up into evening wear, its appearance in artificial light has to be considered.

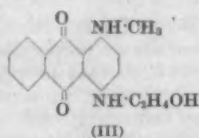
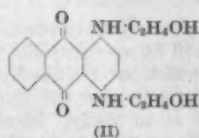
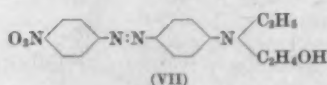
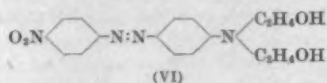
We shall see that it is not possible to combine all these properties in a single dye. Thus, in a general way the more simply constituted dyes of a chemical group are easier to apply but do not possess the highest fastness ratings. Space permits discussion of only a selection of the more important fastness properties and the manner in which they are influenced by the chemical structure of the dye.

Fastness to Light

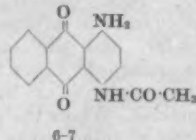
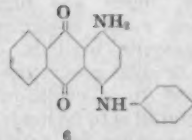
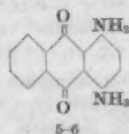
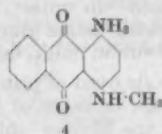
To the ultimate user, resistance to light and to washing are perhaps the most familiar fastness properties expected of dyes. With the advent of materials composed in part or wholly of the newer longer lasting fibres, many of which are most easily dyed with disperse dyes, the search for dye structures with the highest possible resistance to both agencies has been increasingly pursued.

There appear to be no consistent relationships between the chemical structure of a disperse dye and its fastness to light. The fibre itself also plays a part. With a few exceptions, for example, fastness ratings, compared with dyeings on cellulose acetate, are lower on nylon and about the same on Terylene.

The effect of a given chemical group can be seen in certain pairs of dyes, but it is evident that the position of the group as well as its nature is a determining factor. The adverse influence of a second aliphatic hydroxy group is shown in the following two pairs of dyes, where VII and III are faster to light than are VI and II respectively—

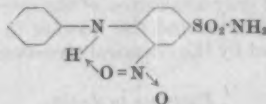


Again, from the series shown below it would appear that fastness to light improves as the basic nature of the amino group is diminished, but such relationships do not apply generally—

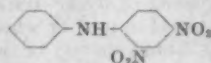


(The figures are light-fastness ratings (B.S. 1006) of a medium-depth dyeing on bright cellulose acetate.)

It has been suggested⁴¹ that a nitro group in the *ortho* position to a secondary amino group confers good light fastness because of the possibility of hydrogen bonding, thus—

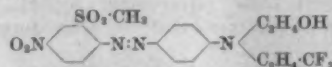


but a much lower fastness is exhibited by the similar compound—

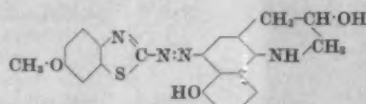


in which the same possibility exists. Enhancement of light fastness by replacing nitro groups *ortho* to

the azo link by CF_3 and $\text{SO}_2\cdot\text{CH}_3$ has been observed⁴². The same authors show that fluorine in the coupling component has a similar effect, providing that at least two fluorine atoms are present on the second or third carbon atom from the nitrogen in an *N*-fluoroalkyl group. By combining both effects excellent light fastness is obtained, as in the red dye—



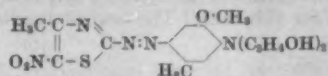
The dyer draws almost all his blue disperse dyes from the anthraquinone class. While in most respects they are satisfactory, they have two drawbacks. Firstly, they are, with the exception of one or two modern ones, sensitive to the action of burnt gas fumes. This will be discussed later. Secondly, they are not dischargeable and hence cannot be used, except in very pale shades, where discharge effects are to be printed on to the dyed ground. Thus much effort has been expended in attempts to produce a satisfactory blue azo dye. This can be done by using heavily substituted azo components, or better if the substituents form external hetero rings, as in⁴³—



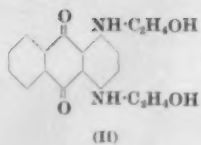
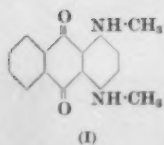
Celliton Discharge Blue 5G

Unfortunately, all these examples have a fastness to light of the order of only 2-3.

There has recently appeared, however, a dischargeable blue, the light fastness of which approaches that of the anthraquinone blues and to which it represents a serious challenge. It is Eastone Blue GFD, which probably uses as first component a derivative of 2-aminothiazole. Using the unsubstituted 2-aminothiazole—which is made on a large scale for the production of one of the sulpha drugs—German chemists⁴⁴ prior to 1939 produced a number of disperse dyes, which, however, did not appear to offer any advantage over existing types. Compared with the corresponding azo dyes prepared from 2-amino-6-methoxybenzothiazole, they were found to be yellower, not so fast to washing, and much less resistant to light. By introducing a nitro group into the thiazole ring, chemists of Eastman Kodak⁴⁵ found that the resulting 2-amino-5-nitrothiazole, when diazotised (with difficulty) and coupled with derivatives of *N*-hydroxyalkylanilines, gave blue disperse dyes with a light fastness considerably greater than that of existing types. An example of such a dye is—



The appearance of a blue dye reasonably fast to light, fast to burnt gas fumes, and capable of being discharged represents a real advance. It provides dyers with a direct means of obtaining blue



Mixture
 $\frac{I + II}{2}$

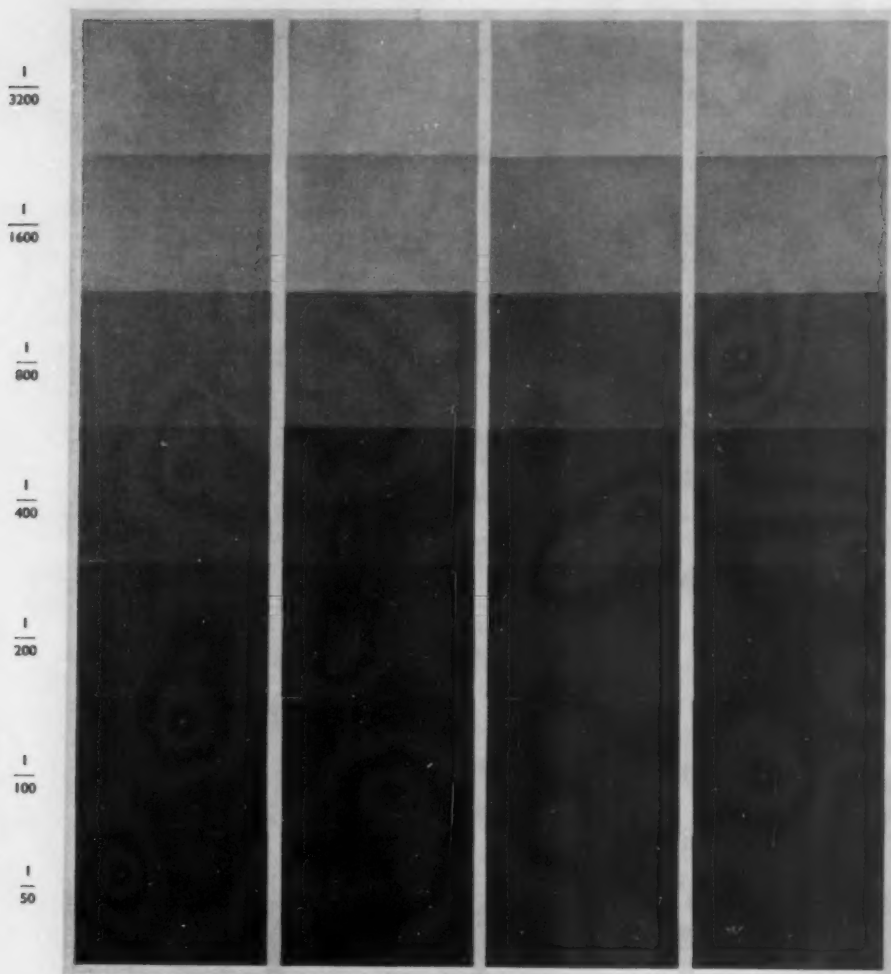
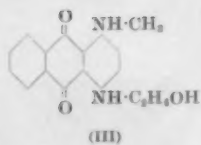


FIG. 1—Additive Behaviour of Mixtures of Disperse Dyes
(Secondary cellulose acetate dyed from a 40:1 soap (2 g./litre) bath at 80°C. for 1 hr.)
(The figures at the sides of the patterns give the amounts of dye used in the bath, in gram-molecules per 100 g. of cellulose acetate)

2% Serisol Fast Brown BTN

1.35% Serisol Fast Yellow 5GD
1.00% Serisol Fast Red 2B
0.29% Serinyl Blue 2G

Normal Dyeings (1 hr.)

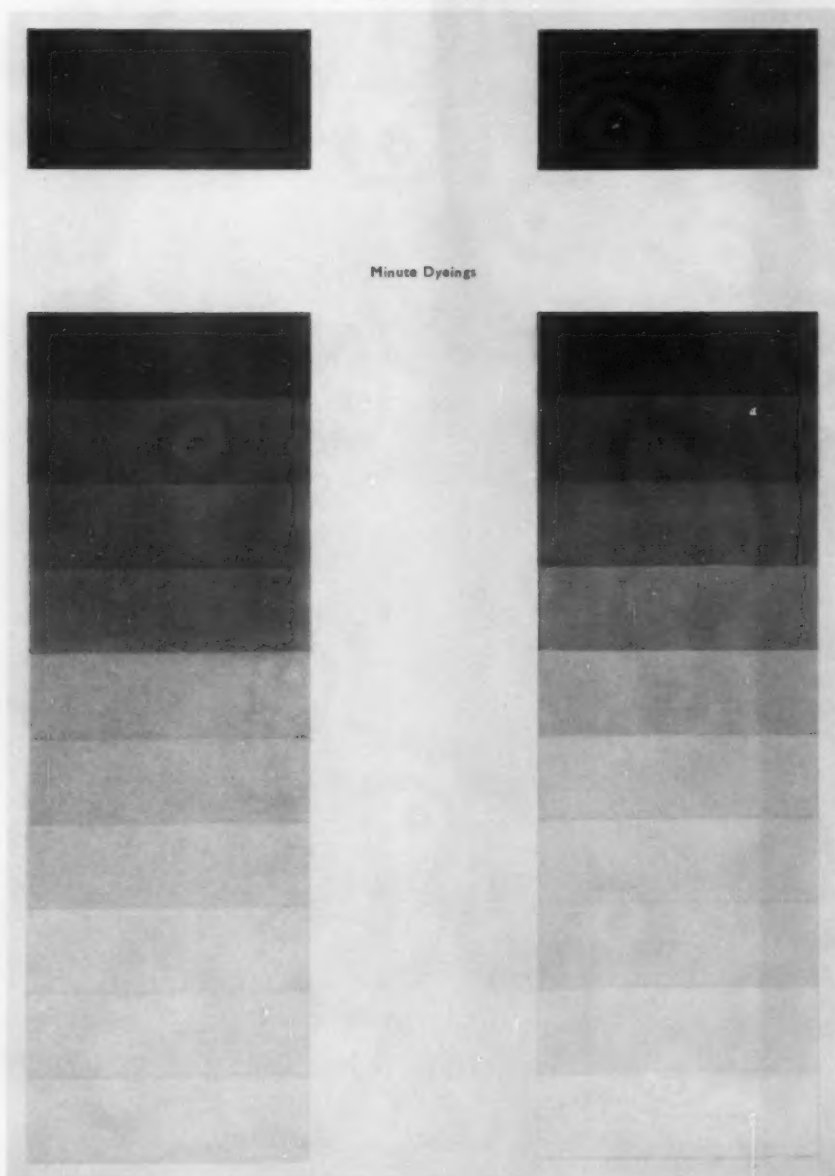


FIG. 2—Jig-dyeing Properties of Mixtures — Effect of Varying the *Components*
(Secondary cellulose acetate at 80°C. "Minute dyeings" successively dyed for 1 min. each in same dyebath)

0.55% Serisol Brilliant Blue BG
0.45% Serisol Fast Yellow GD

0.96% Serisol Brilliant Blue BG
0.24% Serisol Fast Yellow GD

Normal Dyeings (1 hr.)



Minute Dyeings



FIG. 3—Jig-dyeing Properties of Mixtures — Effect of Varying the *Proportions* of the Components
(Secondary cellulose acetate at 80°C. "Minute dyeings" successively dyed for 1 min. each in same dyebath)

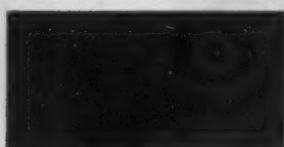
2% Serisol Fast Brown BTN

1.35% Serisol Fast Yellow 5GD

1.00% Serisol Fast Red 2B

0.29% Serinyl Blue 2G

Normal Dyeings (1 hr.)



Minute Dyeings

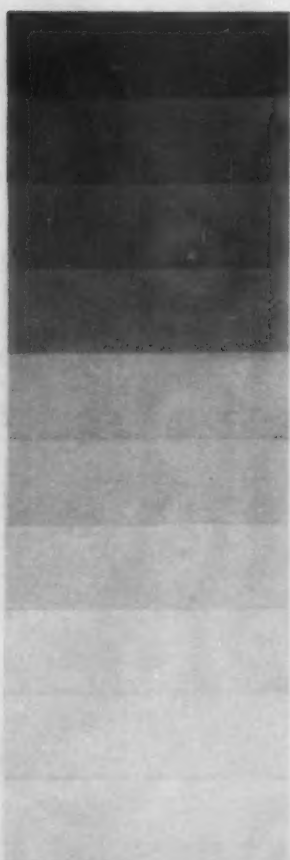


FIG. 2—Jig-dyeing Properties of Mixtures — Effect of Varying the Components
(Secondary cellulose acetate at 80°C. "Minute dyeings" successively dyed for 1 min. each in same dye bath)

0.55% Serisol Brilliant Blue BG
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0.96% Serisol Brilliant Blue BG
0.24% Serisol Fast Yellow GD

Normal Dyeings (1 hr.)



Minute Dyeings



FIG. 3—Jig-dyeing Properties of Mixtures — Effect of Varying the Proportions of the Components
(Secondary cellulose acetate at 80°C. "Minute dyeings" successively dyed for 1 min. each in same dye bath)

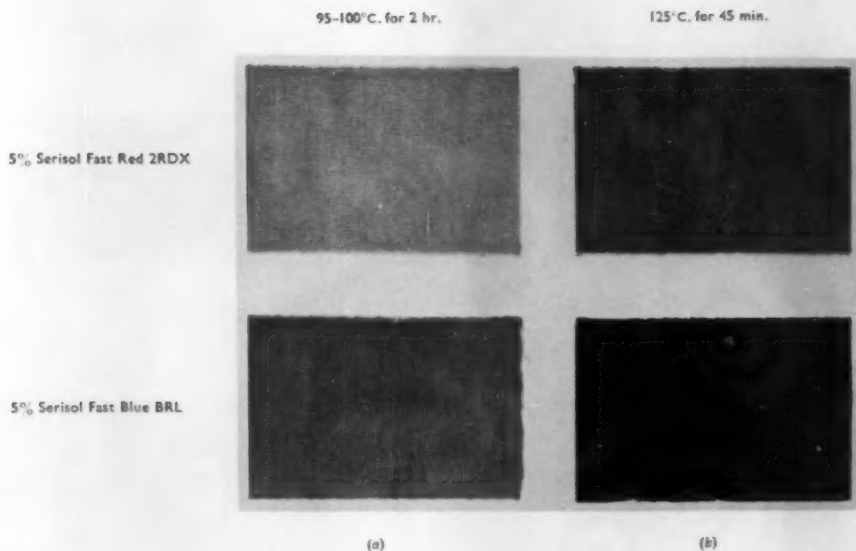


FIG. 4—Non-subliming Dyes on Terylene — Effect of Temperature of Dyeing

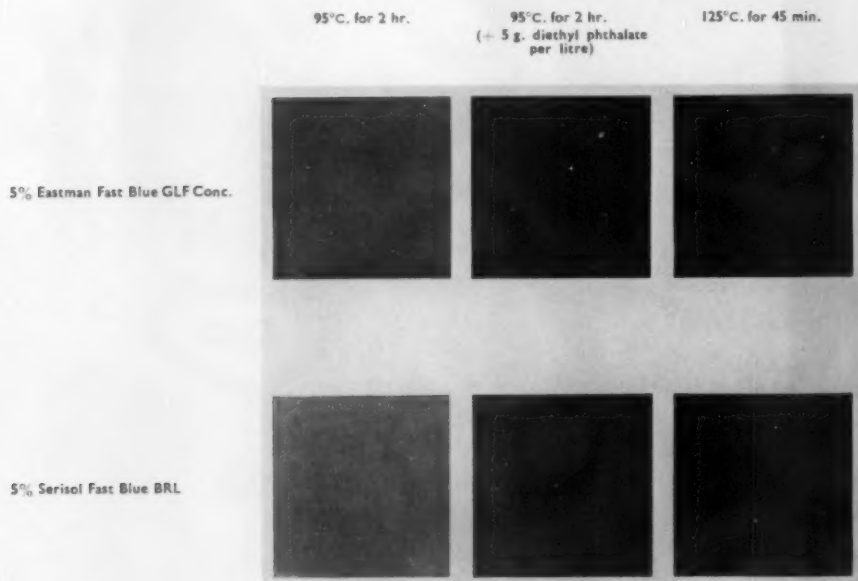


FIG. 5—Slowly Diffusing Dyes on Cellulose Triacetate—Effects of Diethyl Phthalate and Temperature of Dyeing

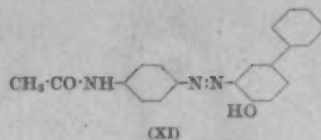
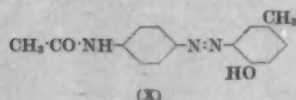
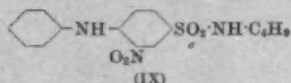
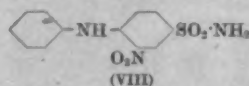
dyeings on secondary acetate, which require no aftertreatment with a gas-fume-fading inhibitor. Unfortunately, fastness to light is lower on nylon and triacetate. It will be valuable, also, to printers, who will now be able to add to the very limited number of ways of producing discharge styles on secondary acetate with medium and dark blue grounds. Greater scope will thus be given to a type of work already stimulated in recent years by the introduction of thiourea dioxide (Manofast (Hardman & Holden)). This reducing agent discharges most disperse azo dyes under neutral and weakly alkaline conditions, without harmful effect to the fibre.

Mention must be made of a weakness shown by certain yellow aminoazo compounds. This is the rapid and reversible change in hue known as phototropism, which used to be a source of trouble, but is now rarely met with, since the offending dyes are no longer offered by dye manufacturers. Phototropy has been shown⁴⁶ to vary in speed and degree with temperature, being non-existent at temperatures of -80°C . and below, and not to occur on certain substrates, e.g. cellulose nitrate. The generally accepted explanation of this phenomenon is that the dye undergoes a *cis-trans* change of structure on exposure to light, the two forms differing from one another in hue.

Fastness to Washing and to Water

As might be expected, fastness to wet treatments of disperse dyes, as with other classes, follows the "easy on—easy off" principle. That is to say, where good fastness is required, the dyer can expect to have to take more trouble, either by selecting dyes which are more difficult to apply, or by using one of the more elaborate dyeing methods. An example of this is afforded by comparing the wash fastness of a given dye on various fibres which differ in the ease with which they are dyed. Thus faster results are obtained on Terylene dyed above 100°C ., or on cellulose triacetate dyed at 100°C ., than on secondary acetate dyed at 80°C .

Fastness to wet treatments improves with increasing molecular complexity. It follows, therefore, that yellows as a class are less fast, because they are structurally the simplest of the disperse dyes. Where the molecule can be increased in size without too much effect on the hue, improved fastness is obtained, but at the cost of dyeing and building-up power. Thus in the following pairs of yellow dyes, IX and XI are faster to washing and water than are VIII and X respectively—

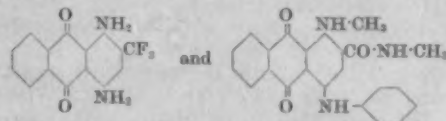


Fastness to Burnt-gas Fumes

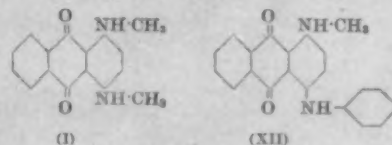
All dyes of cellulose acetate are familiar with what is usually referred to as "gas-fume fading". The dyes which are susceptible to this fault include anthraquinone blues, violets, and reds, which contain primary or secondary amino groups, and also certain yellows and oranges containing primary amino groups. The same dyes on hydrophobic fibres are much more resistant to gas fumes and only very infrequently give rise to complaints.

The fading of susceptible dyes on cellulose acetate was shown by Rowe and Chamberlain⁴⁷ to be due to the presence of oxides of nitrogen, which diazotised the primary amino groups and nitrosated the secondary amino groups of the dye. Couper⁴⁸ has since indicated in a study of the fading of 1:4-bismethylaminoanthraquinone that the reactions which occur are more complex, and that the end-products of fading are similar to those which are formed during fading by light.

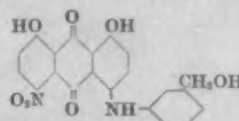
Resistance to the action of oxides of nitrogen is improved by reducing the reactivity of the amino or substituted amino groups present, as has been demonstrated by Salvin and his colleagues⁴⁹. Two ways of doing this are—(i) to have in the *ortho* or related position to the amino group an electronegative substituent, as, for example, in the dyes—



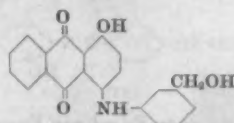
or (ii) to have present arylamino instead of amino or alkylamino groups. Thus Duranol Blue 2G (XII) is less readily affected by burnt gas fumes than Duranol Brilliant Blue G (I)—



Unfortunately, both devices give rise to inferior dyeing properties, but it is possible to improve these somewhat by putting into the molecule a group which is known to assist the dyeing of cellulose acetate. Such a group is hydroxyalkyl, used with discrimination. Modern fume-fast blues such as Eastman Fast Blue GLF are no doubt built up on these lines and have structures related to⁵⁰—

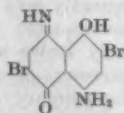


Similar is Latyl Violet B (DuP)⁵¹—



It is possible to push the reduction of the reactivity of the amino group too far and by giving the dye weakly acidic properties to introduce other undesirable features, such as instability to alkali during or after dyeing.

Another approach is to use an alternative structure, as in Artisil Direct Blue GFL, which is probably⁵²—



This is an interesting example of the resurrection of an early used type of compound, naphthaquinoneimine.

Yet another method of minimising the gas-fume nuisance is to use the more readily applied sensitive dyes and protect them by applying an inhibitor, i.e. a compound which preferentially absorbs the oxides of nitrogen. This may be a water-soluble substance padded on, such as triethanolamine, which is removed at the first washing and which possesses the disadvantage of reducing the light fastness of many direct cotton dyes which may be present on cellulose acetate-cotton or cellulose acetate-viscose rayon mixed fabrics. Alkaline salts, e.g. borax, do not have the latter defect, but they, too, are lost on washing. A more satisfactory solution is to use an inhibitor which is substantive. Products of this kind are available, and are exemplified by *NN'*-diphenylethylenediamine (Duranol Inhibitor GF (ICI)), *NN'*-diphenylacetamide (Anti-Acid 3546 (BrC) and Protac 30 (YDC)), and GF Inhibitor BASF. While these, particularly the last two, confer valuable protection, it must be remembered that they do so by expending themselves. Hence there is still room for an inhibitor having greater activity and substantivity⁴⁹.

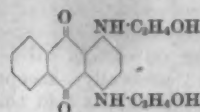
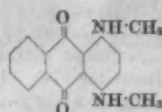
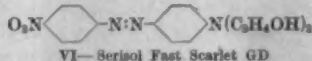
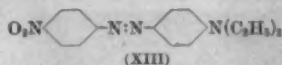
More recently, another type of fading of some disperse dyes on the cellulose acetate and polyester fibres has been reported⁵³. This occurs in absence of oxides of nitrogen and is thought to be due to ozone; hence the description *O-fading*. Unhappily, the gas-fast blues typified by Eastman Fast Blue GLF are susceptible. Gas-fume inhibitors, however, protect against this too, as do anti-oxidants, e.g. *p*-octylphenol.

Fastness to Sublimation

Reference to one other fastness property is made because of the prominence given to it by the growing use of heat-setting treatments after dyeing. The newer fibres such as Terylene and cellulose triacetate are often used either alone or in combination with other fibres for the production of fabrics which undergo thermal finishing operations,

e.g. pleating. The dyes used for such fabrics should possess good fastness to sublimation, otherwise loss in depth or change in shade of the dyeing and marking-off on to other fibres or pleating paper may occur. Good fastness to sublimation is important also for printing.

Apart from the value of a large molecule, the only other structural feature which can be beneficial in promoting stability to heat in a disperse dye is the hydroxyl group, particularly when attached to an alkyl radical. This can be seen in the following examples, in which XIII and I mark off, while VI and II are fast, when triacetate dyed with them is thermally finished either dry at 200°C. or with steam—



In the early days of Terylene, before dyeing with carriers and at temperatures above 100°C. had been explored, it was found that the hydroxyalkyl group so diminished the affinity of a dye for Terylene as to make it of no practical value. This was unfortunate, because such dyes included just those which, being non-subliming, were of special interest for the new fibre. The development of these two dyeing methods resolved the dilemma by enabling some of these faster types to be readily applied. Fig. 4 compares the results obtained with two examples by applying them (a) at 95–100°C. for 2 hr. and (b) at 125°C. for 45 min.

From this short discussion of the relation between chemical structure and a few of the more important of the properties expected of a disperse dye two conclusions can be drawn—(i) as stated at the outset, no single dye can possess all the ideal properties required for a given purpose; (ii) the dyes with superior fastness characteristics are also those whose application by hitherto conventional methods is the least satisfactory. But the durable newer fibres and modern requirements demand these better properties and compel attention to be given to the finding of methods by which the faster dyes can be effectively used. This will be discussed next.

THE APPLICATION OF DISPERSE DYES

Secondary Cellulose Acetate

The discovery of the disperse technique not only gave the dyer a means of dyeing cellulose acetate: it provided him with a simple and direct means as straightforward as, though in general slower than, the application of direct dyes to cellulosic fibres. As a consequence, little or no change in the general routine and equipment of the dyehouse was necessary. Since disperse dyes did

not provide the wet fastness usually sought in yarn-dyed goods, they were, and generally still are, applied to cellulose acetate piece goods on the winch or the jig according to the type of fabric. Some yarn is, however, dyed with disperse dyes when it is intended for fabrics finished in the loom state.

In the early days of cellulose acetate its thermoplastic property and its property of delustering at temperatures above 85°C. led to practical difficulties, particularly in dyehouses accustomed to handling cotton. It was soon realised that cellulose acetate fabrics required processing with more care. Machinery manufacturers have also improved equipment, e.g. jigs, which give the dyer more control over preserving the physical characteristics of the material. Even so, the problem of *moiré* on certain qualities of cellulose acetate fabric dyed on the jig has not been completely solved.

In common with that of other hydrophobic fibres, the dyeing of cellulose acetate on the jig suffers from certain disadvantages. Because of the low water absorption, cooling on the jig rolls, where much of the dyeing takes place, occurs more rapidly than with hydrophilic fibres such as cotton or viscose rayon. Unless, therefore, the dyes being used dye at roughly equal rates between, say, 60° and 80°C., i.e. have good temperature-range characteristics, listing troubles may result. Unfortunately, dyes which are suitable in this respect include many of lower fastness, particularly to washing. In order to take advantage of the faster types, it is necessary to reduce the cooling during dyeing, e.g. by covering the jig. It is of interest here to observe that the defect of poor temperature range properties can be turned to advantage. The small regular particle size of many of the commercial types enables disperse dyes with such properties to be used in cake dyeing and other machines with liquor circulation. The dyer, by careful temperature regulation, can control the rate of absorption of the dye and thus obtain uniform coloration throughout the mass.

Much effort has been expended, especially in America, on attempts to speed up the dyeing of cellulose acetate with disperse dyes. Since temperatures higher than 80°C. are precluded, the aim has been to use solvents and swelling agents, of which, perhaps, aqueous alcohol and ammonium thiocyanate respectively have been most successful⁵⁴. Such methods are subject to limitations imposed by, for example, the difficulty of finding dyes of sufficient solubility in the solvent used, the cost of recovery or loss of solvent, and the risks of fabric damage attendant on the use of swelling agents.

Disperse dyes reserve or only slightly stain cellulosic fibres, and this property enables mixed fabrics, e.g. cellulose acetate-viscose rayon, to be dyed in contrasting cross-dye effects or in solid shades by simultaneously applying appropriate direct dyes to the viscose rayon.

Nearly all the coloured cellulose acetate produced is disperse-dyed. It comes to the wearer in many different forms, e.g. the linings of suits, costumes, or coats; ladies' dress fabrics such as taffetas, satins, and grosgrains; and knitted or woven

lingerie fabrics. It is also widely used in combination with other fibres such as viscose rayon, cotton, or wool for the production of a large variety of dresswear and furnishing fabrics.

Polyamide Fibres

Nylon was the first of the truly "synthetic" fibres and was found to be readily dyed with disperse dyes. Prior to its discovery these dyes were practically entirely used for the coloration of cellulose acetate. Disperse dyes have provided and still provide the simplest and most direct way of dyeing nylon and related fibres. They possess the property of covering yarn irregularities better than any other class of dye, including acid, metal-complex, and chrome dyes. Another useful property is that individual dyes are compatible with one another, so that preferential absorption of certain dyes in admixture does not occur.

Disperse dyes are applied to nylon in a similar manner to that used for secondary acetate, although generally at somewhat higher temperatures, e.g. 85°C. The fastness to light of these dyes on nylon as compared with that of the same dyes on cellulose acetate varies from dye to dye. In general, anthraquinone blues possess very similar fastness on both fibres; whereas azo oranges, scarlets, reds, and crimsons have inferior fastness on nylon. Azo yellows usually possess similar fastness to light on the two fibres, although diphenylamine yellows are generally less fast on nylon.

Burnt-gas fumes do not normally cause fading of sensitive disperse blues, violets, etc. on nylon, though the reason for this is not understood. According to Couper⁴⁹ it is not due to the primary amino (end) group in the fibre molecule.

The fastness to washing of disperse dyes on nylon is generally slightly less than that of the same dyes on secondary acetate and considerably inferior to that of acid, metal-complex, and chrome dyes. Nevertheless, the washing fastness is adequate for many requirements, e.g. nylon stockings, pastel shades on lingerie fabrics, and light to medium shades on dress goods.

In general, nylon piece goods are set prior to dyeing on suitable stenters or hot-cylinder machines, and the necessity for selecting dyes with good sublimation fastness does not, therefore, arise.

The introduction of nylon gave fresh stimulus to the search for disperse dyes with an improved order of fastness properties that would match the greater durability of the fibre itself. The demand for such dyes has continued to grow with the introduction of still newer fibres—the polyester, acrylic, and triacetate fibres.

Polyester Fibres

It was obvious in the early days of the polyester fibres that, even though the disperse dyes ought theoretically to be suitable, in practice too few of them dyed sufficiently well to form a range possessing satisfactory fastness properties. As has already been stated, the trouble lay not in the power of Terylene to adsorb disperse dyes but in the rate (of diffusion) at which it was able to do so. It was not long before means of increasing this rate were evolved and applied.

A simple method not requiring special equipment consists in dyeing in the presence of a "carrier". Very many organic substances will act in this way, but for health, economic, and other reasons the number used in practice is limited to not more than a dozen, of which diphenyl and its 2- and 4-hydroxy derivatives have been widely used in this country; and benzoic and salicylic acids in America. The proportions which are effective vary widely from substance to substance, but they are generally much less than the quantities associated with the action of swelling agents. In practice the device has many defects. Thus, there is with some of the recommended carriers a risk of producing uneven or speckly dyeings, or the light fastness may be affected.

As mentioned earlier, the use of temperatures of 120–130°C. enables Terylene to be successfully dyed in all depths with a range of dyes which either dye too slowly or require a carrier when applied at the normal boil. High-temperature dyeing possesses many advantages over carrier methods. Apart from avoiding the complication of adding to the dyebath something which either melts easily, smells, is volatile in steam, or is difficult to wash off later, the high-temperature method is more rapid, dyes more level, and produces results which are, for certain dyes, slightly faster to light and washing⁵⁵.

These facts have stimulated much interest in dyeing at these elevated temperatures, and British, Continental, and American machinery manufacturers have designed and produced machines capable of withstanding the necessary pressure (up to about 25–30 lb./sq.in.). Machines of the liquor-circulating type suitable for dyeing loose stock, slubbing, or yarn have been particularly successful, and many are at present being used for dyeing Terylene in these forms. The piece dyeing of Terylene fabrics at temperatures up to 120–130°C. presents more problems, although pressurised jigs are now available for this purpose. Although a few of these machines are in operation in this country, piece dyers on the whole have been slow to take them up.

Another machine, of quite different type, for dyeing fabrics at elevated temperatures is the Barotor (DuP). This machine is expensive, more cumbersome than the pressurised jig, and not entirely free from snags in operation. Some work has already been done, too, on the dyeing of Terylene fabrics in beam form in machines of the liquor-circulation type. There is little doubt that many improvements in pressurised machines suitable for piece goods will be made, though at present the carrier technique is mostly used for dyeing Terylene in the piece.

The adoption of temperatures above 100°C. by the dyer has, in turn, called for action on the part of the dye chemist, whose search for suitable new dye structures has included a second look at compounds already tried and found wanting as dyes for cellulose acetate. It has permitted advantage to be taken of the high fastness properties of dyes of e.g. the Latyl (DuP) range, whose building-up powers, even so, are limited.

In general, the disperse dyes on Terylene possess adequate fastness to washing, water, sea water, chlorinated water, and perspiration and are unlikely to be affected by burnt-gas fumes. Selected dyes possess sufficient fastness to sublimation to withstand thermal finishing operations, e.g. pleating. At present there is a lack of a comprehensive range of dyes combining good building-up powers with a fastness to light adequate for furnishing fabrics.

The coming of Terylene has already had an effect, which was perhaps only partly anticipated, on the size and the direction of the demand for disperse dyes. Up to its introduction, by far the larger proportion of these dyes went into ladies' apparel and linings, which were mostly piece-dyed in pale to medium shades by silk and rayon dyers. A considerable proportion of Terylene is no doubt destined, both alone and in admixture, for men's wear, traditionally dyed to heavy shades in loose form or as slubbing. An interesting consequence of this is that many dyers in the wool textile areas are having their first real experience of disperse dyes. In their efforts to cope with increasing quantities of the newer man-made fibres some of them have also been among the first to install and use the machinery specially designed for working above 100°C.

Cellulose Triacetate

This is the latest of the new fibres, hardly yet, as a commercial entity, two years old. It has appeared in this country under the names Courpleta (Courtaulds Ltd.) and Tricel (BrC), and in America as Arnel (Celanese Corp. of America) and Trilan (Canadian Celanese Ltd.). It is interesting to wonder what part the invention of the disperse dye technique may have played in preventing an earlier development of this fibre, by encouraging its less hardy relation, secondary acetate. The provision of an easy means of dyeing the latter was a good complementary reason for choosing it in spite of its weaknesses, for its manufacture at that time posed fewer problems. A satisfactory solvent for the triacetate was not available in large quantities, and this must have been much regretted, since this completely acetylated cellulose is not so readily saponified and delustrated, and was, as made then*, more readily dyed with basic dyes. If the Ionamines and the disperse dyes had not come along till some time later, might not the efforts to find a satisfactory method of spinning the triacetate have been much intensified, have ultimately succeeded, and have resulted in the abandonment of the secondary type?

It is said⁵⁶ that the present-day availability of methylene chloride was an important factor in the reappearance of the triacetate, but helpful too, no doubt, were (i) the desirable property possessed by it of taking a permanent set when treated at a suitable temperature and (ii) the willingness and the interest shown in recent years by dyer and by dyemaker in coping with the problem of colouring the flood of new fibres.

* 1914—known as *Lustron* (The Lustron Co., U.S.A.).

Disperse dyes provide the simplest and most direct way of dyeing cellulose triacetate, and its affinity for them lies between those of secondary acetate and of Terylene. As, however, it can be dyed at the boil, most disperse dyes will produce medium shades, and selected ones can be used for full shades. Cellulose triacetate has been hailed in some quarters as not requiring assistants or temperatures above the boil for its coloration, and for many shades this is true, but to obtain full shades with certain slowly diffusing dyes, which possess desirable fastness to sublimation and light, the use of one or other of these devices is necessary. This applies in particular to blue dyes. As assistants or swelling agents diethyl phthalate⁵⁷ and tripropyl phosphate⁵⁸ emulsified in a sulphated fatty alcohol and used at a concentration of 5 g./litre have been shown to be effective. Dyeing at 125°C. enables similar results to be obtained in a shorter dyeing time. Fig. 5 gives an indication of the increase in depth that may be obtained with Eastman Fast Blue GLF and Serisol Fast Blue BRL by using these dyeing methods.

Because of the slower rate of diffusion of disperse dyes in triacetate as compared with secondary acetate and also because of its lower water imbibition, covered jigs are recommended for the dyeing of flat triacetate fabrics. With open jigs it is difficult to obtain medium to dark shades in reasonable dyeing times.

In general the fastness to light of disperse dyes on triacetate is slightly less than on secondary acetate, whereas the fastness to washing is superior, although not so good as on Terylene. Dyes which are affected by burnt gas fumes on secondary acetate are at least as sensitive on triacetate, although the fastness can be improved by certain of the inhibitors previously mentioned.

Disperse dyes are largely or completely removed from triacetate by dry-cleaning with trichloroethylene. Perchlorethylene and white spirit, however, are quite safe, and most disperse dyes possess good fastness to these solvents. Steps are already being taken to replace trichloroethylene with perchlorethylene in dry-cleaning plants which use this type of solvent.

Heat setting of triacetate fabrics will usually be carried out after dyeing, as presetting reduces the affinity for disperse dyes to such an extent that the production of medium to dark shades would not be a practicable proposition. Dry-heat setting can be effectively carried out at about 180°C. for a short time. Setting under these conditions does not greatly influence the fastness of disperse dyes, although if the somewhat severe condition of 210°C. for 2 min. recently reported⁵⁹ is used the fastness to washing is improved. A range of hues which possess adequate fastness to post-setting and other thermal finishing operations such as pleating is available.

Acrylic Fibres

Of the acrylic fibres, Orlon 42 and equivalent products can be dyed with disperse dyes to pale to medium shades of satisfactory fastness. The saturation value of disperse dyes on Orlon 42 is

considerably less than on secondary or tri-acetate, nylon, or Terylene, and for this reason the use of carriers or high-temperature methods of application, although helpful, is not so effective as in the dyeing of Terylene. Disperse dyes are generally preferred to basic, acid, or vat dyes because of their relative ease of application and good levelling properties. They are usually applied at or near the boil from a liquor containing a suitable dispersing agent. In general, the fastness to light of disperse dyes on Orlon 42 is at least equal and often superior to that of the same dyes on cellulose acetate. The fastness to washing is very good, being better than on triacetate although not so good as on Terylene.

Most of the Orlon 42 used in this country up to the present has been devoted to knitwear, where its lofty, cashmere-like handle is very desirable.

Disperse dyes will not satisfactorily dye Orlon 81 continuous-filament yarn.

Miscellaneous Uses

All disperse dyes stain wool, some fairly heavily, producing dull shades which possess poor fastness to light, washing, perspiration, and rubbing. This property is a nuisance in the dyeing of blends or mixed fabrics containing wool, and necessitates choosing those dyes which stain wool least, using certain auxiliary products such as Dyapol PT (YDC) or Lubrol W (ICI) to minimise the staining, so that protracted dyeing times are required. Advantage has, however, been taken of this weakness. Sheepskin fleeces, which often give "tippy" dyeings with acid dyes, can be dyed quite free from this defect with disperse dyes. Disperse dyes are sometimes used for pale colours on fleeces intended for slipper linings, but they are not recommended for purposes where good fastness to light is desired.

Certain disperse dyes are sometimes used for the surface coloration of plastics such as Perspex (ICI) and cellulose acetate. In order to obtain deep colours, particularly on Perspex, it is necessary to dye in the presence of a swelling agent, and solvents such as butyl alcohol⁶⁰ and Wydecoll (YDC) have been recommended for this purpose.

A limited application of certain disperse dye structures which does not come within the purview of this lecture, because they are used in absence of a dispersing agent, i.e. they are no longer disperse dyes, is the coloration of plastics before moulding. The plastics concerned are cellulose acetate and polystyrene. The Oracet (Ciba) range, for example, contains several members, which are chemically identical with colouring matters used as disperse dyes. They have been chosen for their resistance to the temperatures encountered in moulding (200°C.) and are marketed without dispersing agent in the form of finely ground powders.

SUMMARY AND PROSPECT

The discovery of the disperse dye technique provided an illustration—perhaps the first—of the axiom that the commercial success of a new fibre depends on finding a satisfactory method of colouring it. This addition to the dyer's resources enabled advantage to be taken of the valuable properties of cellulose acetate rayon, not the

least important of which is its lustre, the quality given to cotton by the process whose discovery this lecture commemorates. The debt owed by cellulose acetate to the disperse dyes has been well repaid, for the success and increasing use of the fibre has in its turn ensured a position of importance for this class of colouring matter. This importance has been much increased in recent years with the coming of the man-made fibres, whose long-lasting qualities have underlined the need for greater fastness. The search for this fastness, to judge by the considerable patent literature, has so far confined itself to the improving of existing types. Such improvements, particularly when they result in better fastness to light and washing, are often accompanied, as we have already seen, by diminution in dyeing power.

Fortunately, these same newer fibres have provided the means of utilising certain of the faster dyes by compelling consideration to be given to recently developed methods of application, which speed up the dyeing process. Many dyers have already installed modern equipment or have suitably modified existing machinery for this purpose, and there are made and used today disperse dyes whose dyeing properties would have put them in the "laboratory curiosity" class a few years ago. Methods of application based on the principle of first placing the dye on the fibre by padding, and following with exposure to heat, during which dyeing proper takes place, are of this type, and have the added advantage of allowing disperse dyes with widely differing dyeing rates to be applied together. The Pad-Roll process⁶¹, which uses infrared as the source of heat, is an interesting example of such methods. A novel system being developed by the British Rayon Research Association passes the padded fabric through a fluidised bed of hot sand. This promising idea would appear to have something in common with some of the catalytic processes of modern organic chemical industry and provides an illustration of the usefulness of studying, where possible, the techniques of other branches of industry.

From such considerations it is safe to predict that dyer, machinery maker, and dye chemist together will take the development of these more complex disperse dyes to the limit, though it is doubtful whether the combined demands of colour and fastness will thereby be fully met. The continuing interest in "mass dyeing" of certain of the hydrophobic fibres, with its attendant disadvantages, is an indication that the disperse dyes at present cannot provide adequate fastness for every purpose.

What other possibilities are there? Interest is still being shown in a principle that was used in the fairly early days of cellulose acetate. This is the use of solvents in the dyebath, which cause a certain amount of swelling of the fibre and so admit dyes which have little affinity when applied in the ordinary way. Such dyes, as has been shown already, are often of the faster types, and an acceptable method using solvents would therefore assist in achieving better fastness. Recently from Leeds has come news⁶² of another approach to the

use of solvents in the dyebath, which, if applicable to the disperse dyes, may lead to the same end. Aftertreatment, widely practised in applying direct cotton and acid dyes, has not, so far, been successfully applied to the disperse dyes. One exception—the Eastofix (Eastman Kodak) range of selected dyes, which are aftertreated with metal salts—is of limited value owing to the considerable shade change caused by the aftertreatment. Formation of the dye on the fibre from simpler components, which could be applied as dispersions, offers most attractive possibilities, though, in order to facilitate matching, the process of synthesis would need to be less complicated than the method by which azoic combinations are developed. Evidence that such possibilities exist is provided by the formation of a phthalocyanine in the Phthalogen Brilliant Blue IF3G (FBy) process⁶³. Another approach is to incorporate in the dye molecule chemical groupings, e.g. the 1:3-butadiene residue⁶⁴, which provide a method by which the dye, once on the fibre, can be polymerised by simple means and without significant change of shade.

Short of the discovery of a new chromophore with the right properties, it is not easy to visualise the means by which perfection can be achieved. Meanwhile disperse dyes are being called for in increasing variety and quantity, as one after another new fibre makes its appearance. With this increase come new problems, requiring for their solution a combination of those qualities of intellect, skill, and versatility which are found together in one man only when he happens to be the sort of genius whose name and work we are honouring tonight.

* * *

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The Importance of Temperature and Relative Humidity in Light Fastness Testing

K. McLAREN

Differences in temperature during exposure are unlikely to be a direct cause of anomalous results in light fastness testing; differences in effective humidity, however, are of extreme importance and can cause variations of as much as four grades. The effective humidity during daylight exposure in Great Britain by the method specified in *B.S. 1006 : 1955* has an average value of 20%. Fading lamps should be operated at the same value: the effective humidity in certain lamps is too low and cannot be increased; in others it is too high when running under the makers' recommendations, but in such cases the preferred value can be easily obtained.

INTRODUCTION

In Europe, the statement that a dyed or printed textile material has a light fastness of 4 means that, when it is exposed to daylight facing south at 45° under glass, its rate of fading is very similar to the rate of fading of the internationally accepted light fastness Standard 4 exposed simultaneously under exactly the same conditions. This method, which in principle has been employed in Europe since 1914¹, has one serious limitation—the time required to produce the necessary amount of fading on patterns of high light fastness; these might have to be exposed as long as nine or ten months in the north of England before the minimum amount of fading (specified in *B.S. 1006 : 1955*) has occurred. Such a delay cannot, of course, be tolerated in many cases, and therefore the use of fading lamps has become widespread since they were first marketed in 1919.

Although the preferred method of using such lamps is to employ the same stepwise exposure against the same standards as used for daylight testing, it has been found that their use often gives results which are significantly different from those obtained by daylight exposure, and also that

various lamps often differ among themselves, even though all in general use employ the same light source. This subject had been considered in detail², and three possible causes enumerated—

- (i) Differences in spectral composition between arc light and daylight
- (ii) Differences in temperature during exposure
- (iii) Differences in moisture content during exposure.

The first possibility has been investigated experimentally³, and as it accounts for only a small proportion of the anomalies encountered, an investigation into the effect of differences in temperature and moisture content has become essential; both are best studied together, because a change in temperature almost invariably causes a change in moisture content.

THEORETICAL CONSIDERATIONS

It is well known that the rate of fading of some coloured materials varies considerably according to the moisture content during exposure; for example, if pieces of tannin-mordanted cotton dyed with the basic dye Acronol Yellow TC

(C.I. 815) are placed in two sealed bottles, one containing water, the other anhydrous calcium chloride, and both are exposed to light side by side, the pattern exposed in the damp atmosphere will be completely bleached long before there is any detectable fading on the one kept dry. This sensitivity to differences in humidity varies over a wide range, insensitive patterns fading at the same rate at 100% R.H. as at 0% R.H., whilst sensitive patterns have been observed to fade up to sixteen times as fast when damp as when dry.

It has also been found⁴⁻⁶ that the fading rate increases with an increase in temperature providing that the moisture content remains constant. The magnitude of the increase occurring over the range of temperatures encountered in light fastness testing is probably much smaller than that resulting from humidity variations, this view also being held by Hoffmann⁷ and Lead⁸: the greatest variation recorded in the literature is that at 100% R.H. Solway Blue R on wool fades 4.5 times as fast at 140°F. as at 77°F.; at lower humidities the effect is much less, being only 1.7 times at 32% R.H.⁶. Many American workers, however, believe the converse to be true: for example, Norton⁹ states that—

... there are indications that the specimen temperature is perhaps the greatest single contributing factor influencing the rate of fading.

The AATCC Mid-west Section¹⁰ also conclude that—

... in general, all of the [dyed] fibers were affected much more by increases in temperature than they were by increases in relative humidity.

The possible reasons for these divergent views will be discussed later.

It should be remembered that even extreme sensitivity to temperature and/or moisture content is not in itself a cause of anomalies in testing, as, if such changes alter the fading rates of patterns and standards equally, the same result will be obtained: as Lead has remarked⁸—

The use of standards ... allows one to take greater liberties with temperature, humidity, intensity of illumination, etc., for it is always possible that any error will cancel itself out if the standard and the sample being tested react similarly to the particular factor which has not been kept under strict control.

This does not apply to the most widely used American method of testing, which is to determine the time required to produce an easily perceptible fade on the pattern, for here the basic rather than the relative fading rate is being determined, and hence much greater control of exposure is essential.

A few preliminary experiments were made to see whether large discrepancies in test results could be caused by differences in the relative humidity during exposure, and this did in fact occur: exposure against the standards under dry conditions was often found to give results up to three grades higher than those given by an exposure made at high humidities, i.e. 80–85%, and this clearly substantiated the opinion expressed earlier² that this is the chief cause of gross anomalies in testing. The elimination of anomalies arising from this cause is in principle quite simple: all fading lamps should operate at the same temperature

and the same humidity, whose values should be the average of those occurring in daylight testing.

The investigation fell into three sections: Part 1 dealt with the sensitivity of the standards to temperature and relative humidity; Part 2 with the conditions occurring in daylight testing; and Part 3 with the possibility or otherwise of duplicating these values in the various lamps in use at the present time.

1. THE SENSITIVITY OF THE STANDARDS

(a) Sensitivity to Moisture Content

The varying sensitivity of dyed fabrics to changes in moisture content was appreciated in 1928, when, on behalf of the Society, Cunliffe¹¹ joined in the search for light-fastness standards. He naturally concluded that only dye-fibre combinations of low sensitivity could be accepted, and the criterion adopted was that the rate of fading at 100% R.H. should be less than twice the rate at 32% R.H.

The lower figure of 32% R.H. is, however, much higher than that of the air surrounding the pattern in some fading lamps, and the first experiment in the present investigation was to determine the sensitivity of the standards over the complete range of air humidities by the following method—

Pieces of each standard were suspended in 5-lb. glass bottles containing in the bottom silica gel, sulphuric acid solution, or water, which produced atmospheres of constant humidity ranging from 0 to 100% in stages of 20%. The bottles were closed with rubber seals, a small hole was bored in each to prevent pressure building up as the temperature increased, and they were then placed on racks behind the sloping window of a typical exposure house. Exposure was stopped when the standard exposed at 0% R.H. showed a contrast equal to Grey Scale Grade 2 (time did not allow this degree of contrast to be obtained with Standard 7, and with Standard 8 no detectable fading occurred at any humidity even after exposure for two years). The exposed standards were then conditioned, and their reflectance curves plotted on a General Electric recording spectrophotometer. The colour difference between each standard before exposure and after exposure at different relative humidities was then calculated by means of the Adams "chromatic value" formula—

$$\text{Colour difference } \Delta E = 40[(0.23 \Delta V_y)^2 + (\Delta(V_x - V_y))^2 + (0.4 \Delta(V_z - V_y))^2]^{0.5}$$

The results are shown in Table I together with those obtained for two patterns of high sensitivity for comparison, a basic green (Acronol Yellow TC (C.I. 815) plus Acronol Brilliant Blue AN (C.I. 633)) on tannin-mordanted cotton and an azoic red (Brentamine Fast Scarlet R→Brenthol AN) on mercerised cotton; both of these patterns were used in subsequent experiments. These results show that the sensitivity of the standards is very low; visually the amounts of fading at different humidities were even closer than these figures would suggest, and for all practical purposes the fading rates of the standards can be regarded as being independent of moisture content.

TABLE I

Pattern	Colour Difference (N.B.S. units)					
	at R.H.—					
	0%	20%	40%	60%	80%	100%
Standard 1 ...	17.2	19.5	21.6	21.2	24.2	25.5
Standard 2 ...	19.7	23.9	21.1	21.1	23.8	28.7
Standard 3 ...	26.7	22.5	22.0	22.5	24.8	24.3
Standard 4 ...	6.8	6.7	5.9	6.4	7.4	9.2
Standard 5 ...	17.8	14.6	15.8	13.3	14.0	21.4
Standard 6 ...	12.5	10.7	9.5	12.2	10.4	12.6
Standard 7 ...	4.0	3.5	5.1	4.6	4.2	5.0
Basic green ...	12.8	20.0	24.1	30.2	33.9	33.2
Azoic red ...	4.7	8.0	10.2	15.6	25.5	35.5

(b) Sensitivity to Temperature

No work was done on the temperature sensitivity of the standards, as a reliable investigation would have required the construction of special apparatus; this was not considered justified, partly because all the available evidence indicated that the temperature effect was only small and partly because it was known that an investigation into this aspect was being made in the U.S.A., where it is of much greater importance, and duplication of effort was obviously undesirable.

It is worth recording, however, that in routine testing sets of standards were often exposed at various surface temperatures ranging from 50°F. to 180°F., the higher temperature being hot enough to scorch wool patterns. No difference in the spacing was ever observed, which shows that the effect of temperature is similar for each of the standards, and this is, of course, the most important requirement.

2. DAYLIGHT EXPOSURES

Daylight exposures are made behind window glass facing due south at 45°, the patterns and standards being exposed continuously day and night until the required degree of fading has occurred. The temperature and the moisture content of the pattern will therefore vary continuously throughout the exposure period: at dawn the temperature is lowest, the relative humidity of the air is greatest (often reaching 100%), and therefore the moisture content is the maximum; during the day the temperature rises, the humidity of the air consequently falls, and the moisture content of the pattern also falls, reaching a minimum value about 2 p.m.; towards dusk the conditions reverse, and the moisture content returns to its maximum value. It will be noted that these two factors work in opposition as far as the rate of fading is concerned, for when the temperature is highest, the moisture content is lowest, and vice versa.

Apart from these variations in what might be termed the subsidiary factors affecting fading rate, the chief factor, viz. the intensity of the light falling on the pattern, is also varying, as even on clear, cloudless days it varies according to the height of the sun above the horizon and to the angle the incident sunlight makes with the plane of the pattern; in relatively cloudy countries, such as northern Europe, the variations in intensity are, of course, much greater.

It will therefore be readily appreciated that it would be pointless to record the hour-to-hour variations in temperature and humidity during exposure, as the calculation of suitable average values from such data would be quite impossible. The problem was resolved by a very simple method, which consisted in exposing a humidity-sensitive pattern in controlled atmospheres ranging from 0 to 100% R.H. and simultaneously in the normal pattern position. By comparing the amount of fading on the pattern exposed normally with those under controlled conditions the effective humidity in the house could be ascertained quite accurately. The term *effective humidity* is preferred to the more usual term "average relative humidity", as this method takes into account the effects of temperature and light intensity as well. The details of the method are as follows—

A piece of humidity-sensitive material (tannin-mordanted cotton dyed with a mixture of the basic dyes Aconol Yellow TC and Aconol Brilliant Blue AN) was exposed, usually for 24 hr., in each of the constant-humidity bottles already described and simultaneously in an identical bottle from which the base had been removed and which was not sealed; the air surrounding this particular pattern was therefore at the same temperature and humidity as occur in routine testing, and the light intensity was the same as that falling on the patterns in the controlled atmospheres. After exposure the reflectance curves of each pattern were plotted on a recording spectrophotometer, and the reflectance at 620 m μ . (one of the absorption peaks of this green pattern) was plotted against the relative humidity; a typical result is shown in Fig. 1.

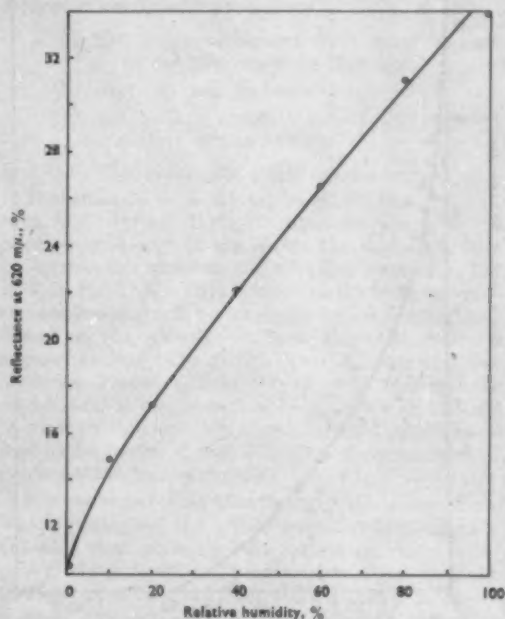


FIG. 1.—Reflectance of Green Patterns after Exposure at Different Humidities

From the reflectance of the pattern exposed in the open bottle, the corresponding "effective humidity" can be read off directly from the graph. On the occasion illustrated in Fig. 1, the pattern in the open bottle had a reflectance of 21%, which corresponds to an effective humidity of 35%.

The effective humidity in the fading house at Blackley (Manchester) was determined on 73 occasions between March 1955 and August 1956 and was found to vary over a very wide range, viz. between 0 and 89%.

It is natural to suppose that the effective humidity would be governed principally by the relative humidity of the outside air, but it is obvious that this cannot be the only factor, because an effective humidity below 10% was found on about twenty occasions, although the relative humidity never fell below 35%. The latter figure is, of course, the *lowest* reached; the *average* humidity during the hours of daylight on that particular occasion was, naturally, considerably higher.

In a previous publication² it was predicted on theoretical grounds that the surface moisture content during exposure would be governed mainly by the proportion of sunshine to total daylight falling on the pattern. If this reasoning were correct, we would expect to find a correlation between the amount of sunshine and the effective humidity, as the surface moisture content is, of course, governed by the effective humidity.

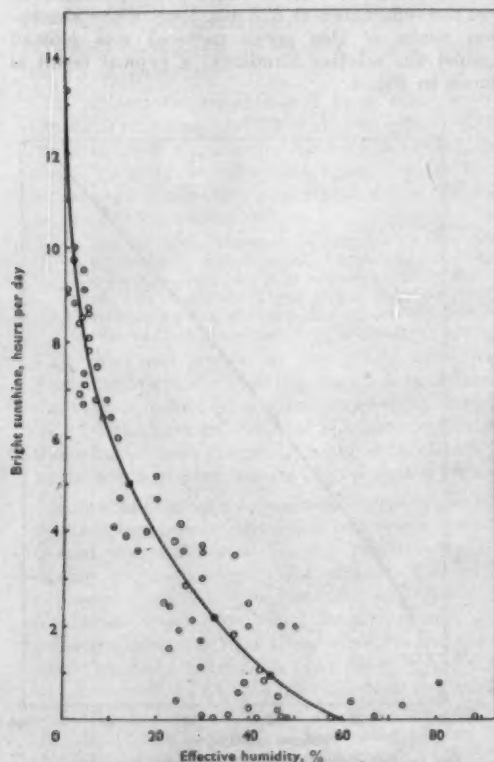


FIG. 2—Effective Humidity as a Function of Amount of Sunshine

The validity of this prediction was therefore tested by plotting the effective humidity against the number of hours of sunshine, this figure being obtained from the daily report published in the *Manchester Guardian*; where a pattern was exposed for more than one day (as at week-ends and in winter), the average value for the whole exposure period was taken. The results are given in Fig. 2, which shows that there is in fact a very close correlation between the two, which is undoubtedly causal.

The reasons which led to the prediction that the effective humidity would be governed mainly by the amount of sunshine were discussed previously², but it is worth repeating them here, partly because they are of such fundamental importance in lamp and daylight testing and partly because further experimental data are now available.

The moisture content (regain) of a textile fibre is governed mainly by the relative humidity of the surrounding air, the temperature of the air having only a slight effect. This is not true, however, when the fibre is exposed to a powerful light source such as the sun or a carbon arc, which is rich in infrared. The radiant energy is absorbed by the fibre and raises its temperature above that of the air mass; the boundary layer of air at the exposed surface is also heated to approximately the same temperature, and in consequence its relative humidity falls; the surface moisture content undoubtedly corresponds to this reduced relative humidity rather than that of the whole air mass surrounding the pattern.

Measurements of the surface temperature are best made with a thermocouple embedded in the fabric surface, and the results of such measurements have been published by Hall¹², Nordhammar¹³, and Boulton and Guthrie¹⁴. A much simpler method, which is probably reliable enough, is to place a thermometer with a blackened bulb on the surface of the pattern fully exposed to the source of radiation, a method now used in the U.S.A. for fading-lamp adjustment⁹. Records of the maximum black-bulb temperature reached each week in the exposure house at Blackley were kept during 1956, the figure actually being obtained from a solar radiation thermometer which automatically registers the maximum value. These results, together with the weekly maximum shade temperature of the outside air and of the air just behind the glass in the fading house (measured with the bulb shielded) are given in Fig. 3.

The reduction in relative humidity which will occur when the air is heated at the surface of the pattern can be obtained from a psychrometric chart, and it is clearly adequate to explain the low effective humidities actually found. For example, the following values will be typical of a sunny day in summer—

Average air temperature	... 70°F.
Average relative humidity	... 50%
Average surface temperature	... 140°F.

When the outside air is heated up to 140°F., its relative humidity will fall to 5%.

This type of calculation also indicates that the effect of the relative humidity of the outside air

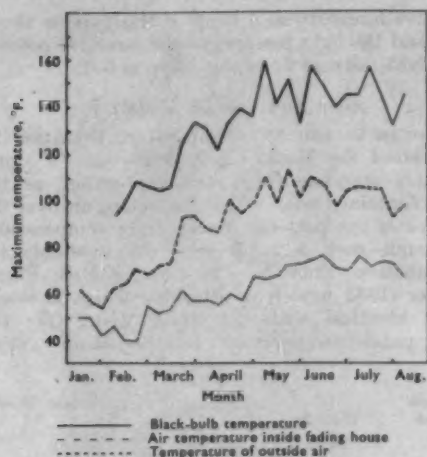


FIG. 3—Weekly Maximum Temperatures

is only slight in sunny weather, as even if it had been 100% instead of 50% in the example just quoted, it would still have fallen to 10% at the surface of the pattern.

Of course, when the amount of sunshine is low, the effect of the relative humidity becomes of much greater significance; this undoubtedly accounts for the greater amount of scatter in the region of low sunshine totals in Fig. 2.

The amount of scatter at higher sunshine totals was actually very much less than was expected: obviously the effect of a given number of hours of sunshine would be lowest when it occurred very early or very late, and greatest in the middle of the day; similarly, the time of year also would be expected to be significant. But the amount of scatter shows that these differences, as well as the differences in relative humidity, are insignificant.

Preferred Effective Humidity

It is obvious that, as the prime function of a fading lamp is to give the same result as a daylight exposure but in a much shorter time, the effective humidity should be equal to the mean value occurring in daylight testing in the United Kingdom. The calculation of the mean value of the 73 measurements recorded would not necessarily be a reliable figure even for Manchester: to repeat the measurements in several other places would have been difficult, but the close correlation between the amount of sunshine and the effective humidity suggested that the effective humidity corresponding to the average amount of sunshine occurring in the United Kingdom would be a suitable value.

The arithmetic mean of the daily average of bright sunshine for the 187 stations for which data are readily available¹² was therefore calculated and found to be 4.0 hr.; the effective humidity corresponding to this figure is 20%. The range of average daily sunshine totals in the British Isles is not excessive, being from 2.9 hr. for Manchester to 5.2 hr. for Jersey; this difference was found to be relatively unimportant as far as anomalies in daylight testing are concerned, and

details of this investigation will be published shortly.

Preferred Temperature

The preferred surface temperature for fading lamp operation will naturally be the average value occurring during daylight exposure, weighted to allow for the varying intensity of light falling on the pattern. Although such a value would be extremely difficult to obtain, all the evidence indicates that the actual value will not be important providing that it is somewhere near the mean, and it was felt that a preferred value could be specified. The most important criterion is that it should be less than the maximum value occurring in daylight, not so much because of its effect on the fading rates of patterns and standards, as because it may cause thermal discoloration of some of the newer synthetic fibres. Taking everything into consideration, therefore, it was thought that the preferred value should be $120 \pm 10^\circ\text{F}$.

3. THE CONDITIONS IN FADING LAMPS

Now that preferred values of temperature and effective humidity have been established, it is necessary to study the actual conditions in each type of fading lamp in general use in the United Kingdom to see how far they depart from the preferred values and whether a closer approach can be obtained by adjusting the controls provided by the lamp manufacturers.

The determination of the temperature and the effective humidity in fading lamps is much simpler than in daylight exposures, because the conditions in a lamp are constant, and therefore the complicated indirect method of determining the effective humidity is no longer necessary: it can be obtained from a psychrometric chart providing that the following conditions are known—

- (i) the temperature of the mass of air within the exposure chamber
- (ii) its relative humidity
- (iii) the surface temperature of the exposed surface of the pattern

and each of these can be easily measured.

This method is, however, based on the assumption that during daylight exposure the average relative humidity of the air at the surface of the pattern is the same as the effective humidity, i.e. 20% in the U.K. This is not strictly true, because surface heating will occur in the sealed bottles, and therefore the average surface humidity will be somewhat less than 20%; there is some indirect evidence, however, that the effect of this is only slight, and it can therefore be ignored. However, in view of this and other assumptions which have had to be made, it was felt that some method of confirmation was essential.

It is perhaps fitting that the original observations which prompted the whole investigation, namely the fact that anomalies in testing do occur, also provided the method of confirmation, for if the light fastness of a humidity-sensitive pattern is determined by exposing it at different effective humidities, the results will vary; if the predicted

conditions in a lamp are correct, the result will be the same as from a daylight exposure.

The pattern used was mercerised poplin dyed with the azoic combination Brentamine Fast Scarlet R→Brenthol AN, whose light fastness varied from 3 when the effective humidity was 100% to 6-7 at 0%. This pattern had been exposed to daylight at different times and in different parts of the United Kingdom, and its light fastness was usually 5; details of this work will be published shortly. It was not affected by the excess of ultraviolet radiation emitted by the enclosed carbon arc, so that if the predicted lamp conditions are correct, the same result will be obtained.

effective humidity in a lamp of this type is about 2%, and the light fastness of the sensitive pattern (daylight fastness 5) in this lamp is 6-7.

Atlas Fade-Ometer Model CV

In order to reduce the temperature, the Atlas Co. introduced the Model CV in 1928, the "V" presumably standing for forced ventilation, as the lamp contained a fan which, by forcing air over the surface of the patterns, reduced the temperature. Although such a lamp was not available for examination, there is a modified K.B.B. Fugitometer (1932 model) at Blackley which is essentially identical with the Atlas Model CV; the black-panel temperature in this lamp varied

TABLE II

Maker	Model	Fig.	Years when Marketed	Arc Wattage	Humidifier	Heat Filter
Atlas Electric Devices Co. Inc. (Chicago, U.S.A.)	Fade-Ometer C	4	1919-1928	1700	None	None
	Fade-Ometer CV	5	1928-1932	1700	None	None
	Fade-Ometer FDA	6	1932-1941	2000*	Wet wicks	None
	Fade-Ometer FDA-R	7	1941 (current)	2000*	Wet wicks	None
Calico Printers Asscn. Ltd. (Manchester)	Fading lamp	9, 10	1934 (current)	1000	Heated water tray	Water screen
Kelvin, Bottomley & Baird Ltd. (Glasgow)	Fugitometer	—	1924-1928	1500	None	None
	Fugitometer	8	1928-1942	1750	Saturated salt soln. or water	None
	Fugitometer	11	1945-1953	1750	Heated water tray	Water screen
Kelvin & Hughes (Industrial) Ltd. (Glasgow)	Fugitometer	12	1953 (current)	2000 (max.)	Atomiser	Water screen

* Owing to improvements in electrical design, the FDA-R model, although of identical wattage, is actually more powerful than the FDA model

The lamps which were studied are listed in Table II and illustrated in Fig. 4-12; they are all based on the enclosed carbon arc (the latest type of lamp, based on a xenon arc, was received only after the work had been completed). Many of these lamps have been installed at Blackley, and daily measurements of the black panel (surface) temperature have been made since February 1955. These readings provided most of the data for calculation purposes; in two cases measurements were made in other laboratories, and in another the conditions had to be inferred.

Atlas Fade-Ometer Model C—K.B.B. Fugitometer 1924-1928 Model

These lamps were the first to be marketed and were as simple as possible, consisting merely of an enclosed arc surrounded by a non-rotating frame-holder. The arc not only emits actinic radiation which causes fading, but, like the sun, it emits infrared radiation as well; in fact, it is even richer in infrared radiation than is sunlight, 75% of its radiation being in the region above 1400 m μ . compared with 30% for sunlight¹⁶. Black-panel temperatures in a lamp of this type were always between 138° and 174° F., the higher temperature being high enough to scorch the wool of the standards. It is little wonder, therefore, that the original Fastness Committee found this type of lamp to be "quite unsatisfactory on account of the low humidity and high temperature"¹⁷; the



FIG. 4—Fade-Ometer Type C (1919-1928)

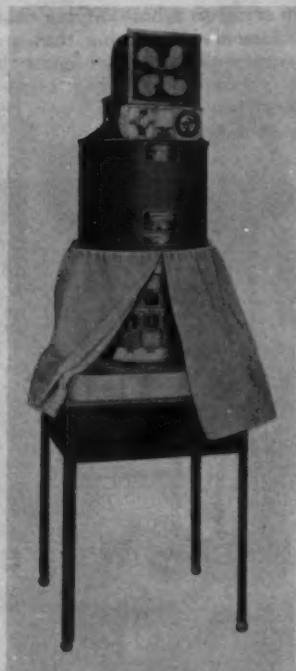


FIG. 5—Fade-Ometer Type CV (1928-1932)

between 94° and 106°r. , which corresponds to an effective humidity of 14% if installed in a laboratory where the air has a relative humidity of 40% at 70°r. The humidity-sensitive pattern had a light fastness of 6-7 in the modified K.B.B. lamp, showing that the effective humidity is still too low to duplicate British daylight exposures.

Atlas Fade-Ometer Models FDA and FDA-R

In these two lamps an attempt was made to increase the effective humidity by passing the incoming air over a series of wet wicks before it entered the exposure chamber. Unfortunately, however, this was not particularly successful, because the surface temperature was much higher than in the CV model, i.e. 165°r. , whilst the air temperature was about the same, 105°r. The effective humidity is therefore very low: even if the air were completely saturated (100% R.H. at 105°r.), the effective humidity would be only 15%, but nothing like complete saturation can be achieved by such a simple method of humidification, and it is doubtful whether values of more than 40% R.H. can be continuously obtained, corresponding to an effective humidity of only 6%. This was confirmed by the observation made on many occasions that the sensitive pattern exposed under the conditions specified by the AATCC in 1954¹⁸ had a light fastness of 6-7, i.e. not significantly different from exposures made in non-humidified lamps.

The low effective humidity in the humidified Atlas lamp was first pointed out by Nordhammar¹⁹ six years ago, and he concluded that "the present

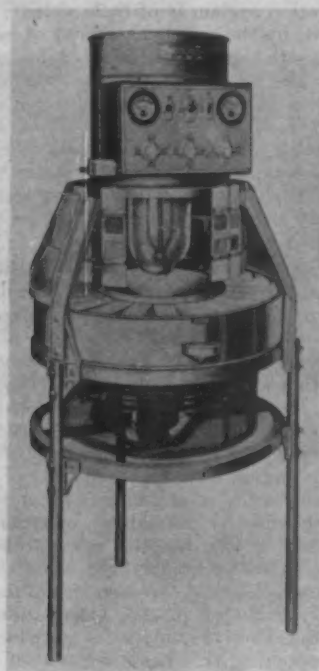


FIG. 6—Fade-Ometer Type FDA (1932-1941)

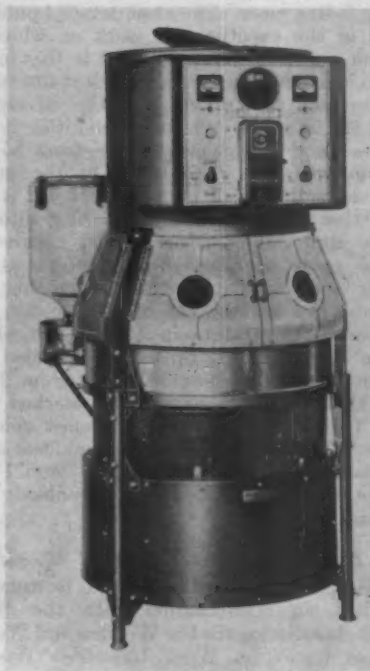


FIG. 7—Fade-Ometer Type FDA-R (1941-)

humidification system is of little value". He also found that, by the simple expedient of inserting a screen beneath the patterns which deflected the air flow over the surface of the patterns instead of round the back, a considerable reduction in surface temperature could be obtained. The Atlas Co. have recently (July 1956) marketed such screens, and the accompanying pamphlet states that "the new AATCC carbon-arc light-fastness test method will specify a recommended black-panel temperature of $145^{\circ}\text{F.} \pm 5^{\circ}\text{F.}$ ".

Whilst these new conditions will result in an increase in the effective humidity, it will still be lower than the preferred value, as this could be obtained only if the air mass had a relative humidity of 70% at 105°F. It must be remembered, however, that the Atlas Fade-Ometer was designed primarily to duplicate the conditions of the AATCC sunlight test, which restricts exposure not only to sunny days in the sunniest half of the year but also to the six hours in the middle of the day, when the intensity of sunshine is greatest. From Fig. 2 the effective humidity corresponding to 6 hr. sunshine is 9%, but this refers to 6 hr. sunshine plus at least 6 hr. daylight when the sun is not shining; such overcast conditions are deliberately excluded in the American test, and therefore the effective humidity is more likely to be that corresponding to a sunshine total of 12 hr. in Fig. 2, i.e. approaching zero.

It is convenient at this point to consider the complete divergence, already mentioned, between American and European workers in this field with regard to the relative importance of humidity variations and temperature variations as factors affecting fading rates. The most detailed published account of the experimental work on which the American conclusions were based is that of the AATCC Mid-west Section¹⁰, and a study of this paper reveals the reason for the divergence. Firstly, the range of relative humidities investigated was not particularly wide—from 35% to 65% compared with 0 to 100% by the present author; secondly, in all their experiments a lamp of the Atlas type was used, i.e. no heat filter and therefore appreciable surface heating occurred, so that the range of effective humidities was even narrower; sufficient data for calculating this range were published for the set of experiments at a black panel temperature of 135°F. , and in this case the 35–65% R.H. range corresponded to a variation in effective humidity only from 9% to 16%, which would not have had a marked effect on the fading rate of even the most sensitive patterns. The range of effective humidities at the other black-panel temperatures (155° and 175°F.) could not be calculated, but they are unlikely to be significantly wider.

K.B.B. Fugitometer 1928–1942 Models

This lamp was designed by Kelvin, Bottomley & Baird Ltd. in collaboration with the British Research Association for the Woollen and Worsted Industries (now the Wool Industries Research Association). The patterns were exposed in separate glass-fronted boxes, each box being

connected in series to a humidifying chamber, the original recommendations being that a saturated solution of common salt be used in the chamber to give a relative humidity of 75%; the use of salt seems to have been abandoned about 1932, water being used instead.



FIG. 5.—Fugitometer (1928–1942)

The surface heating in this lamp was very low, as the fan was most efficient; measurements recently made on the lamp at Torricon* gave black-bulb temperatures of 86 – 102°F. If the air leaving the humidification chamber is at 60°F. and 75% R.H., the effective humidity will be 20%, which is the preferred value. An exposure of the humidity-sensitive pattern in this lamp did give the same result as daylight, namely 5.

C.P.A. Fading Lamp

It has already been mentioned that the enclosed carbon arc emits much more infrared radiation than sunlight, and in 1934 a lamp was marketed which was designed to reduce this by placing an annular water screen between the arc and the patterns, water absorbing infrared radiation very strongly. The effectiveness of this screen is illustrated by the following black-panel temperature measurements, which have been made in one of the two C.P.A. lamps installed at Blackley—

Without screen	154–166°f.
With screen ...	98–104°f.

The air in the exposure chamber in the C.P.A. lamp was humidified by means of a heatable water

* Private communication from Dr. F. F. Elsworth



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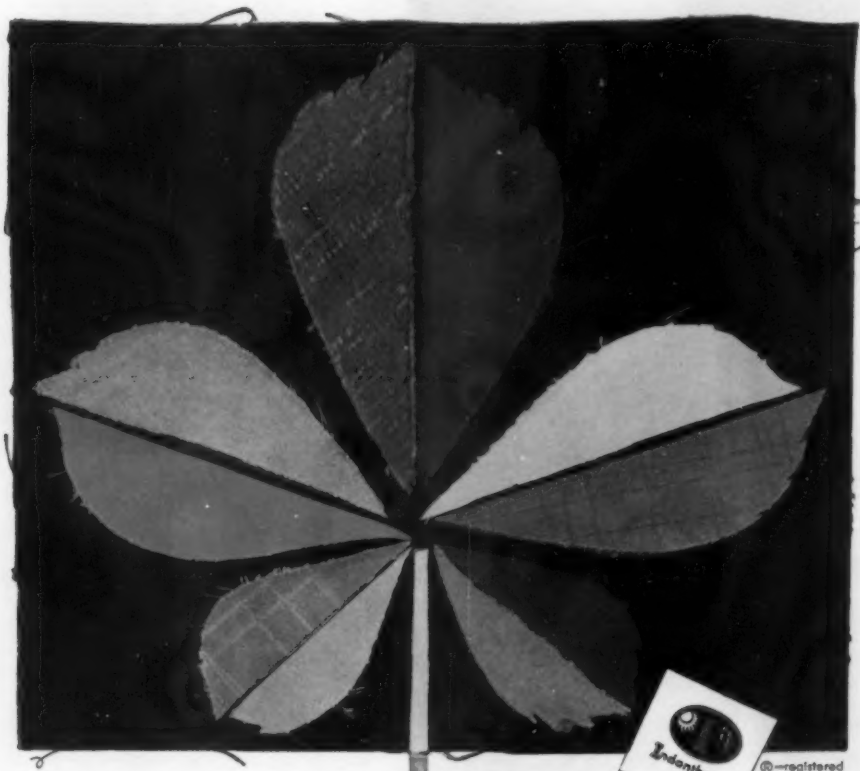
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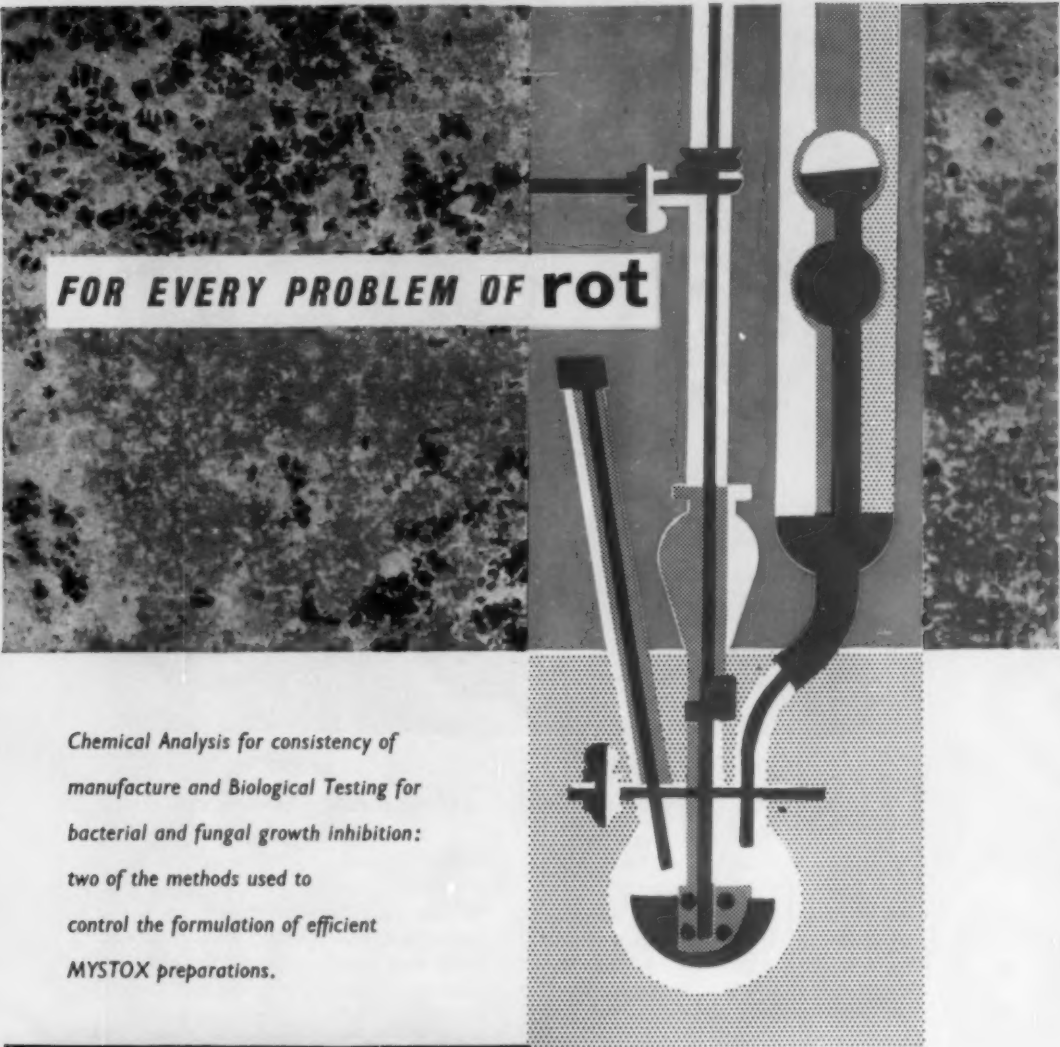
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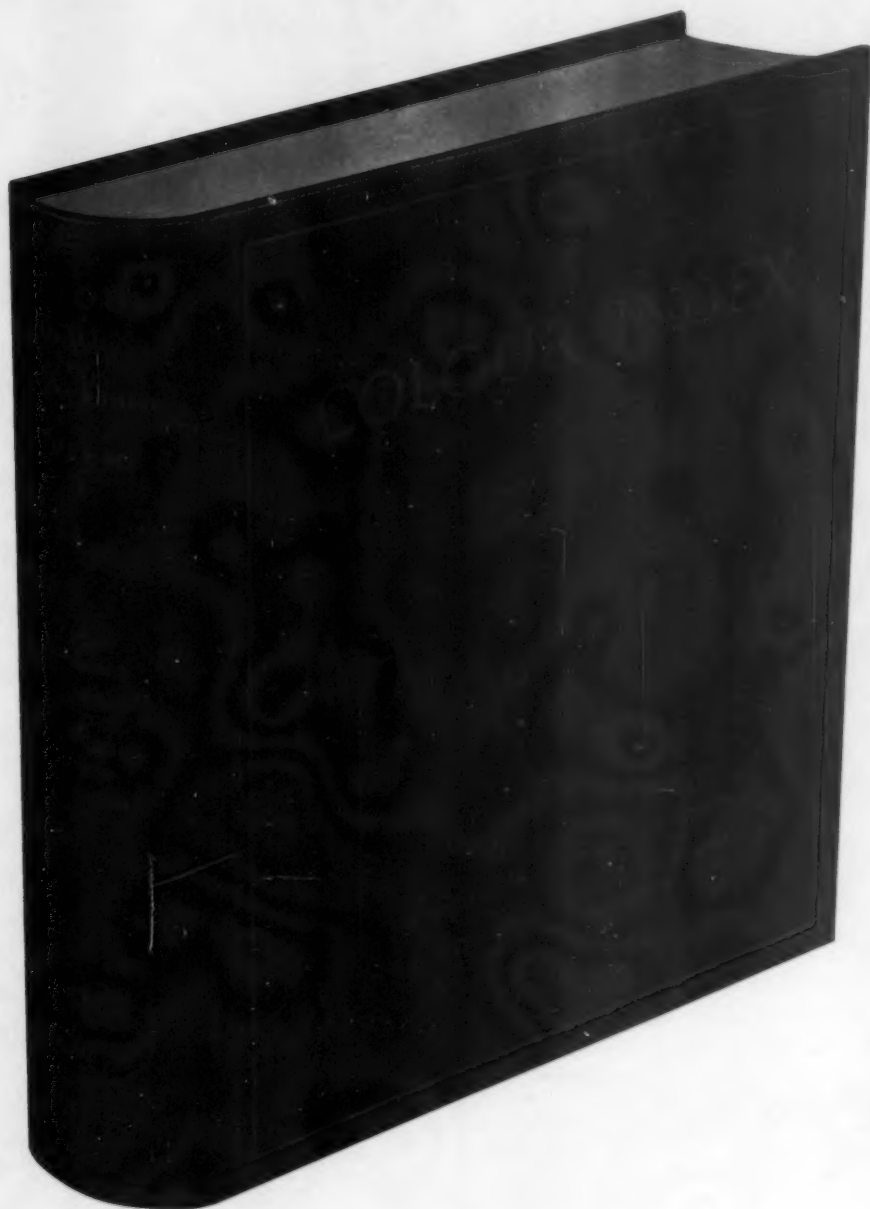
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IMPORTANT*Foreword by the Presidents*

THE first edition of the *Colour Index* was published in 1924. Quickly it became recognised as the standard work on the subject in the English language. Shortly before the Second World War the Society of Dyers and Colourists was planning the preparation of a new edition of the *Index* as many of the dyes listed in it had become obsolete and many new dyes—indeed complete new ranges of dyes—had been introduced since the original publication. The outbreak of the war interrupted these plans and the work could not recommence until 1945.

A *Colour Index* Editorial Panel was set up in 1945 by the Society of Dyers and Colourists and at the same time the American Association of Textile Chemists and Colorists was invited to collaborate in the preparation of the new edition. The two organisations agreed that the work of compiling the new *Index* should be a joint undertaking. In view of the information received from users of the original *Index*, it was decided that it would be advantageous to re-cast it in an entirely new form and to include much more information than was contained in the first edition. Every dyemaker in the world was invited to supply information for the new edition. Many firms collaborated in this way and we acknowledge their help with gratitude. The task of the Editorial Panels proved to be much greater than was anticipated originally and has required of them and their helpers a very great amount of detailed and tedious work. Perhaps it is appropriate that their task should have been completed in the year which marks the centenary of Perkin's discovery of Mauve.

As Presidents of the Society of Dyers and Colourists and of the American Association of Textile Chemists and Colorists we should like to express our thanks to the Editorial Panels and to all who have collaborated with them in the production of these new volumes. We feel sure that future users of the new *Index* will have good reason to add their thanks.

It is recognised that any work of reference will tend to become out of date and it is the intention of the Society and the Association to keep the *Index* up to date by the issue of supplementary volumes as occasion demands.

CLIFFORD PAINE
*President, The Society of
Dyers and Colourists*

RAYMOND W JACOBY
*President, American Association of
Textile Chemists and Colorists*

VOLUME 1 OF THE NEW COLOUR

AN HOUSE 19 PICCADILLY BRADFORD 1 YORKSHIRE

T ANNOUNCEMENT

The Society of Dyers and Colourists

Founded 1884. Sections: London, Huddersfield, Manchester, Midlands, Northern Ireland, Scottish, West Riding of Yorkshire. Junior Branches: Bradford, Leeds, Manchester. Affiliations: The Society of Dyers and Colourists of Australia, The Dyers and Colourists Association of South Africa

*President CLIFFORD FAIRIE D.S.C. F.R.D.C.
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General Secretary J. W. NICHOLLS F.C.I.S.*

DEAN HOUSE 19 PICCADILLY BRADFORD 1 YORKSHIRE

*Telephone
25138-9*

22 October 1956

Dear Sirs

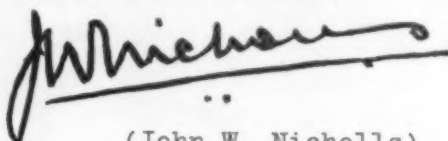
I have been instructed to inform you of the proposals for publishing the SECOND EDITION OF THE COLOUR INDEX which is nearing completion, and to express regret for the delay in publication.

The work is in FOUR VOLUMES and is being published by the Society of Dyers and Colourists and the American Association of Textile Chemists and Colorists.

There have been unforeseen delays in course of preparation, but publication will commence with the delivery of the FIRST VOLUME before the end of this year. VOLUME TWO, if not despatched at the same time, will follow soon afterwards. This to be followed by VOLUME THREE and finally VOLUME FOUR to be published in the early part of 1957.

I am

Yours faithfully



(John W. Nicholls)

GENERAL SECRETARY

To Subscribers

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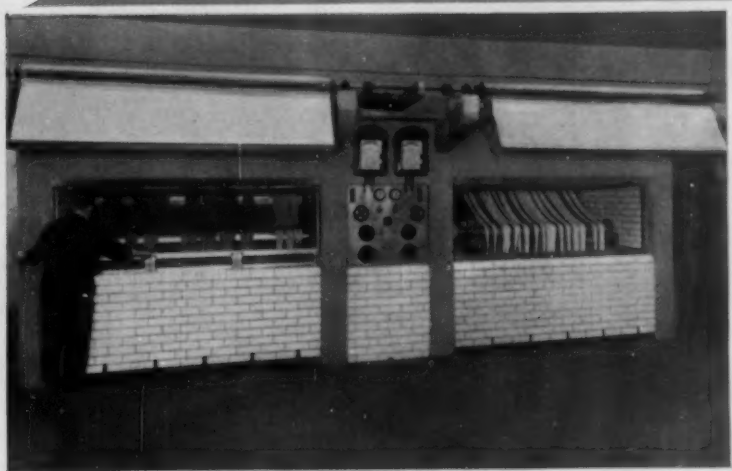
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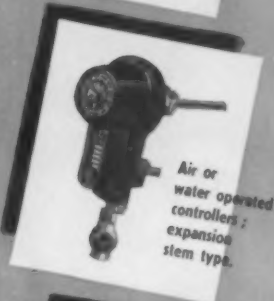
Sir Harold Hartley, K.C.V.O., F.R.S.,
in his Presidential Address to the
Institution of Chemical Engineers.



Drayton type DV3 Controllers installed on Winch
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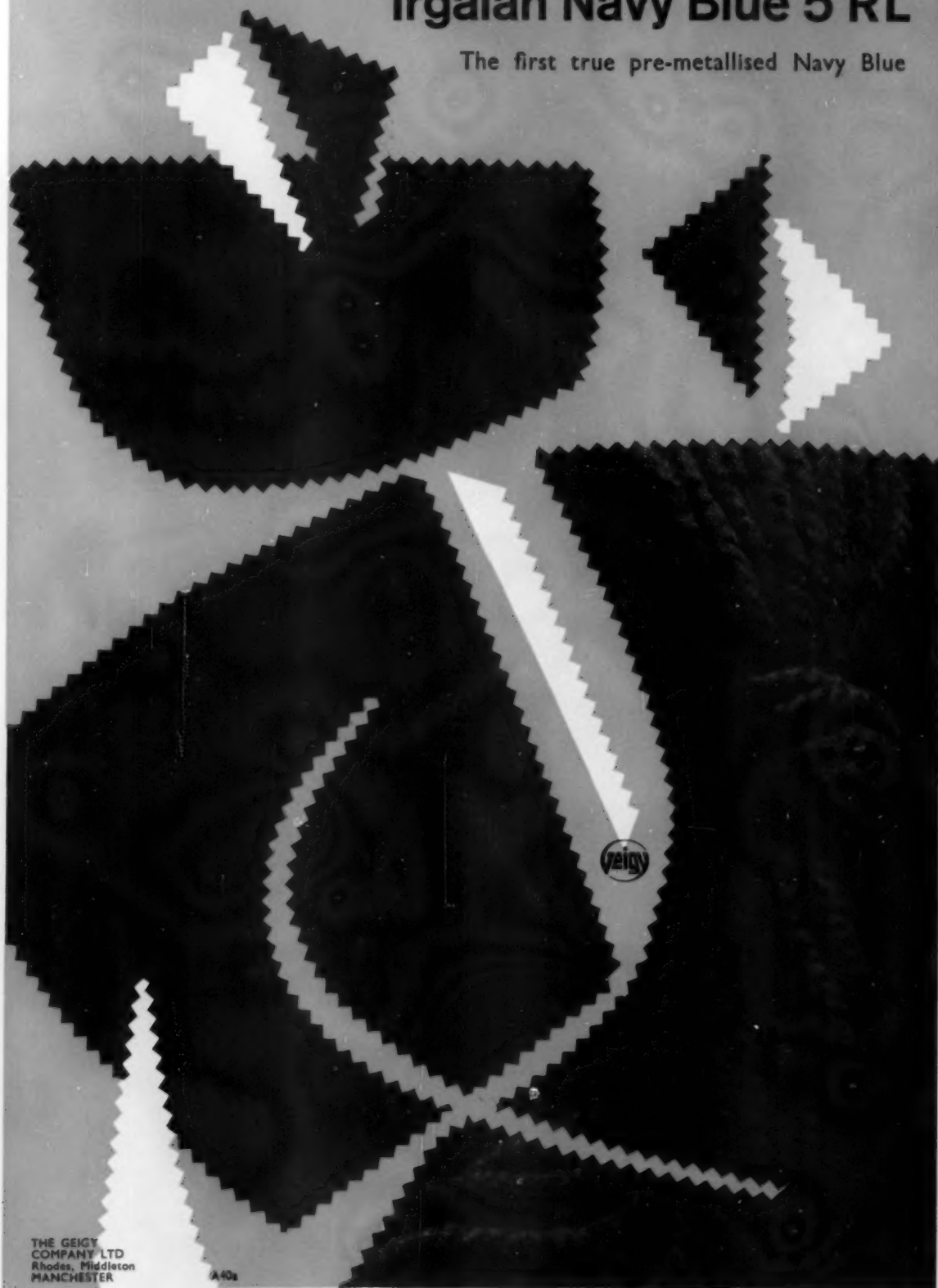
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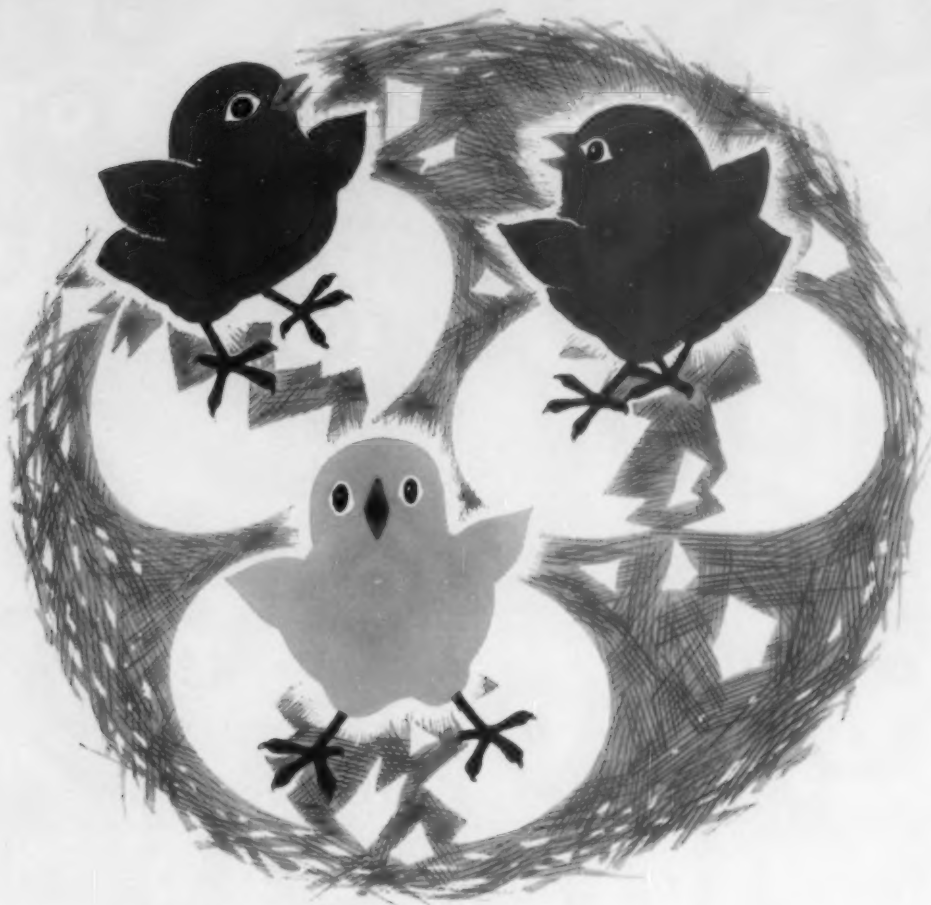
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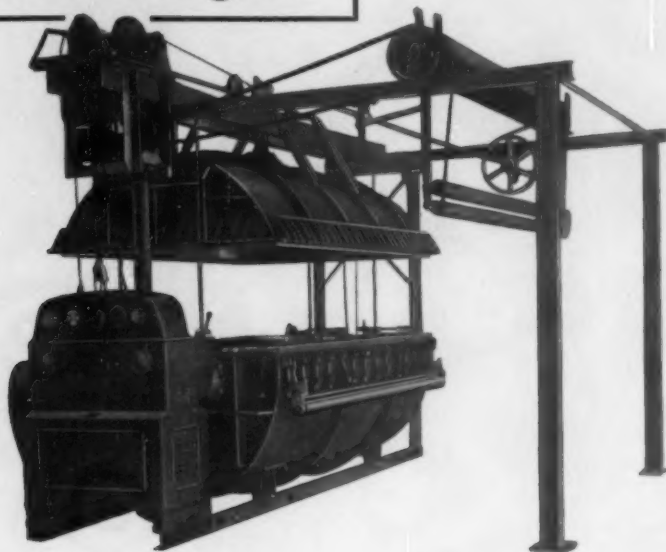
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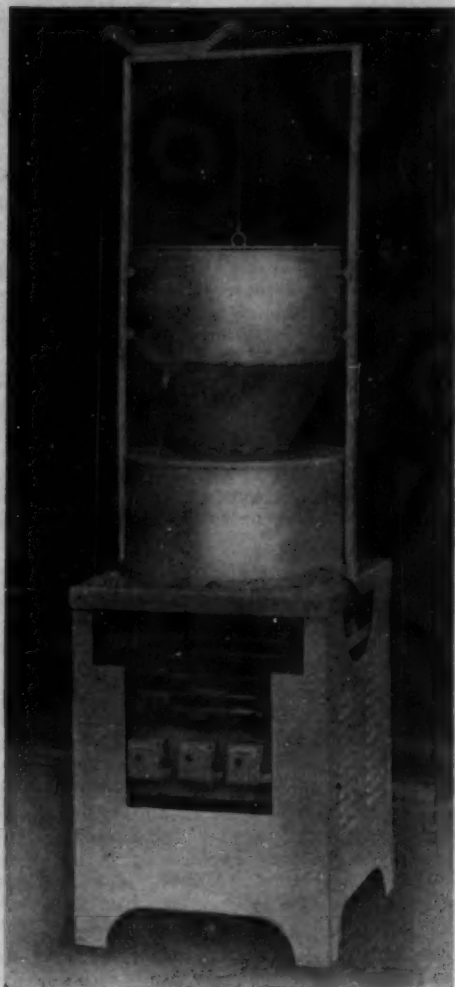


FIG. 9—C.P.A. Fading Lamp (1934-1947)

tray, which formed the base of the lamp; above this tray a fan rotated whose speed could be adjusted. For the first time, therefore, both the temperature and the relative humidity could be varied, the former from 95° to 115°F. and the latter from 50% to 90%. The makers specified certain values of temperature and humidity for imitating various climates, those for the British Isles being 113°F. and 80-85% R.H. As the water screen eliminates most of the surface heating (the black-panel temperature being only about 10°F. higher than the air temperature), the effective humidity is only slightly less than the relative humidity, and therefore the makers' original recommendations result in exposures being made at an effective humidity far higher than can ever occur in daylight testing by the method described in *B.S. 1006*; the humidity-sensitive pattern exposed under these conditions had a light fastness of 3, one grade lower than the lowest value ever found in the daylight test, which was observed during a particularly damp winter.

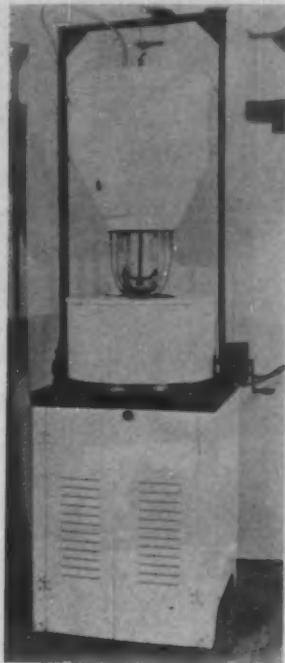


FIG. 10—C.P.A. Fading Lamp (1947-)

The selection of this high value by the makers was prompted by the fact that they had occasionally received complaints that curtains had faded in use, which could have occurred only if the latter had been exposed under conditions of extremely high humidity, at least as high as 80% R.H.⁸ However, such conditions have been used by other concerns for routine testing of all fabrics irrespective of whether they are ever likely to be exposed in use under conditions of high humidity, and this has undoubtedly accounted for many of the wide discrepancies observed between lamp and daylight exposures; when results in this lamp are compared with those obtained in a lamp of low effective humidity, such as the Atlas, differences of as much as four grades can occur.

The preferred effective humidity of 20% can be obtained in this lamp only by emptying the water from the tray, and advice on this matter can be obtained from the makers.

K.B.B. Fugitometer 1945-1953 Model

This lamp resembled the C.P.A. lamp in having a water screen between the arc and the patterns and in having a heatable water tray for humidifying the air in the exposure chamber. Measurements have been made in a lamp of this type through the courtesy of Mr. B. Taylor of Messrs. Fothergill & Harvey (Research & Development) Ltd., and it has been found that the black-panel temperature is about 104°F., some 6°F. higher than the air temperature.

As in the case of the C.P.A. lamp, the desired effective humidity can be obtained only by

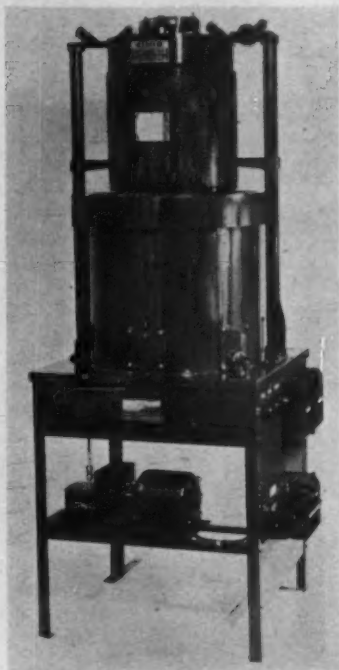


FIG. 11—Fugitometer (1945-1953)

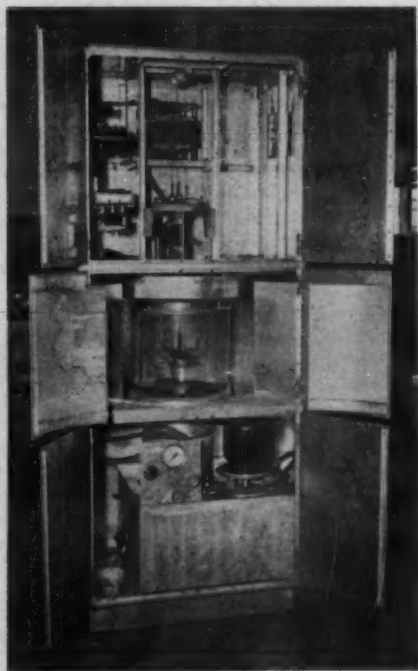


FIG. 12—Fugitometer (1953-)

emptying the water from the tray, and advice on this matter can be obtained from the makers.

K. & H. Fugitometer

In October 1950 Kelvin & Hughes Ltd., the successors to K.B.B., redesigned the Fugitometer and asked the Society's Light Fastness Subcommittee what range of temperatures and relative humidities were required in a fading lamp. At that time it was impossible to give an answer, so the makers decided to make a lamp in which both were independently variable over as wide a range as possible. The lamp contained a water screen, but differed from other screened lamps in two major respects: firstly, humidification was effected by an atomiser instead of a heated water tray; and secondly, the water passing through the screen could be heated. This lamp could therefore be operated at an effective humidity as great as 100%, but in order to duplicate British daylight conditions it should be operated with the heater and the atomiser turned off; under these conditions the humidity-sensitive pattern did have a light fastness of 5.

CONCLUSIONS

The most important cause of anomalous results in light fastness testing is the effective humidity during exposure. In the past the range of effective humidities in routine testing in fading lamps has varied from about 2% in some unscreened lamps to about 70% in the C.P.A. lamp. These differences can cause variations in the results obtained of as much as four grades.

The effective humidity in daylight testing in Great Britain is about 20%, and fading lamps should be operated at the same value. Four carbon-arc lamps are capable of giving these conditions—the three K.B.B. and K. & H. Fugitometers marketed since 1928 and the C.P.A. fading lamp.

Lamps running at lower values, such as the Atlas Fade-Ometers, will give results with sensitive patterns which are higher than those from a daylight exposure made in the United Kingdom and which in extreme cases may be two grades higher; whilst lamps running at higher values, such as were originally recommended in the case of the C.P.A. lamp, will give results as much as two grades lower.

Whilst the preferred conditions for lamp operation will ensure that the results obtained are closest to daylight exposures made in Great Britain, the excess of ultraviolet radiation emitted by the enclosed carbon arc can also cause anomalies, which it is not practicable to overcome. Exposures in carbon-arc fading lamps, even under the preferred conditions of effective humidity, must not, therefore, be regarded as always equivalent to daylight testing, but as a means of obtaining a fairly reliable indication of the actual light fastness when time does not permit a daylight exposure to be made.

* * *

The author wishes to express his thanks to Mr P. R. Dawson, who did most of the experimental

work at Blackley, and to various members of the Society's Light Fastness Subcommittee.

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(Received 13th August 1956)

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- ¹⁴ Boulton, J., and Guthrie, J. C., *J.S.D.C.*, **67**, 190 (1951).
- ¹⁵ Meteorological Office, Air Ministry, *M.O. 572* (London: H.M.S.O. 1953).
- ¹⁶ Cooper, B. S., and Hawkins, F. S., *J.S.D.C.*, **65**, 586 (1949).
- ¹⁷ Report of the Society of Dyers and Colourists on the Work of its Fastness Committee in fixing Standards for Light, Perspiration and Washing (Bradford: the Society, 1934).
- ¹⁸ AATCC, *Tentative Test Method 16A-54*.

ERRATUM

The Behaviour of Formaldehyde-sulphoxylate in Vat Printing (*J.S.D.C.*, **72**, 474 (Oct. 1956))
—In Table III the units in the first column should be **hours**, not "minutes".

Notes

Meetings of Council and Committees

October

Council—3rd
Finance and General Purposes—3rd
Colour Index Editorial Panel—15th and 30th
Publications—16th
Textbooks and Monographs Subcommittee—16th
Perkin Main Committee—19th

Death

We regret to report the loss by death of Mr. J. A. Fergusson (Honorary Treasurer of the Society of Colourists of Australia).

Fourteenth Mercer Lecture

This will be held on Friday, 3rd May 1957, at the Grand Hotel, Leicester, when the subject will be *The Influence of Fibre Types on Dyeing Methods*, and the lecturer Mr. J. S. Ward.

Patents Act 1949

Occasionally inventions are made by persons resident in the United Kingdom and are taken up for development by companies resident abroad. Such companies usually wish to have a patent application filed in their own country. No difficulty arises if a British Patent Application has already been on file for not less than six weeks; but certain steps must be taken to comply with the requirements of the Patents Act 1949 if no British Patent Application has been filed or if such application has only just been filed.

Subsection 5 of Section 18 of the Patents Act 1949 reads as follows—

- (5) No person resident in the United Kingdom shall, except under the authority of a written permit granted by or on behalf of the comptroller, make or cause to be

made any application outside the United Kingdom for the grant of a patent for an invention unless—

- (a) an application for a patent for the same invention has been made in the United Kingdom not less than six weeks before the application outside the United Kingdom; and
- (b) either no directions have been given under subsection (1) of this section or under section twelve of the Atomic Energy Act, 1946, in relation to the application in the United Kingdom, or all such directions have been revoked:

Provided that this subsection shall not apply in relation to an invention for which an application for protection has first been filed in a country outside the United Kingdom by a person resident outside the United Kingdom.

Many inventors seem to be unaware of these provisions. Nevertheless, any person who fails to comply with them becomes liable to serious penalties which can involve both fine and imprisonment. Applications for permission to file patent applications overseas are rapidly dealt with by the Comptroller of Patents and are not refused unless it appears to him that the invention is one of a class notified to him by a competent authority as relevant for defence purposes, e.g. inventions relating to the production and use of atomic energy. No such permission is, however, required if a British Patent Application has been on file for more than six weeks and no direction has been given by the Comptroller of Patents prohibiting or restricting publication of information with respect to the invention.

Inventors who enter into negotiations with persons or companies in overseas countries, even Commonwealth countries, should keep this matter in mind to avoid the risk of getting into trouble.

L.E.J.

Amendment of Canadian List of Food Dyes

The Department of National Health and Welfare announces that Orange I, Orange SS, and

Oil Red XO have been removed from the list of food dyes whose use is permitted in Canada. C.O.C.

Current Food Additives Legislation

The Legislative Service of the Food and Agriculture Organisation of the United Nations in Rome has begun publication of a periodical with the above title, which is intended to report all enactments on intentional non-nutritive food additives published after the end of 1954. The subject index includes the heading "colouring matters", and microfilms or photoprints of any text mentioned can be obtained. The *Bulletin* is issued free.

Colour in Diamonds

The United Kingdom Atomic Energy Authority is prepared to irradiate diamonds on a commercial basis. Diamonds irradiated in a nuclear reactor

become green, which may be changed to brown on subsequent heat-treatment. Particle-accelerating machines may produce a blue or blue-green colour.

Explanations of Theoretical Ideas

The following quotation from the preface to a book *What is Science?* (edited by J. R. Newman. New York: Simon & Schuster Inc. 1955) may have some relevance to the difficulties experienced in explaining and understanding dyeing theory—

Though written for persons without special training, the pieces in this book are not always easy. Neither is modern science. A decent respect for the reader requires that he be told when a subject admits of no further simplification; the contributors have not pretended to explain what cannot be explained. Nevertheless, every effort has been made to speak plainly, to refrain from jargon and nebulous profundities, to enlighten rather than astound. Although some areas are too difficult for any deep explorations, the nature of the problem, the aims of its investigations and the instruments they use have been made clear.

New Books and Publications

Grundlagen der Textilveredlung

Fachteil T 61 aus

Handbuch für Textilingenieure und Textilpraktiker
By M. Peter. 7th edition 1956. Pp. xi + 297.
Wuppertal-Elberfeld: Dr. Spohr-Verlag. Price, DM 9.

This is a completely revised edition of a pocket-manual which aims at presenting the basic principles of dyeing and finishing in a concise and lucid manner to the textile technologist and the textile engineer. Let it be stated at once that Herr Peter splendidly succeeds in this formidable task. The demand for an inexpensive yet authoritative source of information is indicated by the appearance of a seventh edition in just over a decade.

What makes this book so useful and easy to peruse is its clarity, and its economy of words in presenting a survey of the most common dyeing and finishing processes, the chemicals and dyes usually employed, and the machinery required. Throughout emphasis has been placed on the practical side, and there are frequent references to actual processing details, striking evidence of the author's intimate knowledge of the industry. A very useful feature is the inclusion at the end of an appropriate section of a paragraph headed "sources of faults". This should provide an invaluable aid to the dyer and finisher in characterising and, if within his province, preventing similar faults.

The book is divided into 11 chapters and they account for 192 pages out of a total of 312 including advertisements. The remainder comprises an eight-page cross-reference index followed by an extensive bibliography. A supplement of some ninety pages is subdivided into—(i) a directory of German and foreign manufacturers of textile machinery and equipment with emphasis on German sources, (ii) a similar directory of manufacturers of dyes and auxiliaries, and (iii) a very extensive alphabetical list of textile-finishing agents and auxiliaries.

It would be all too easy to pick holes in this text on account of certain simplifications and omissions. But a volume of this nature in order to succeed must necessarily be mercilessly brief and shorn of all trimmings. This inevitably results in a tendency to be dogmatic. It is stated e.g. that the simplest method of assessing colour blindness is by the use of Stilling's colour diagrams. This is a moot point in view of the fact that the Ishihara test is preferred by many. A more valid criticism, however, might be the occasional inclusion of what appear to be blue-prints of machines obtained directly from the manufacturers' drawing boards. Such reproductions are too complex and often hide the essential features amongst a welter of detail. Although this book is completely up to date, the section on the dyeing of polyester fibres is really too brief, even for this kind of text.

The number of misprints does not seem unusually large considering the compass of the subjects presented. On p. 72 "Abbot-Cox" is incorrectly spelt, and so is "Holliday" on p. 73. In the key to the lettering of Fig. 88b on p. 178, a sketch of a compressive-shrinkage machine, *Bf* is given as "Beleuchtungseinrichtung", whereas it should of course be *Befeuchtungseinrichtung* (spray-damper), a confusion of one letter, but what a difference in meaning! The spelling of "bonded fabrics" also requires correction (p. 188).

However, these are very small points and will no doubt be corrected in a subsequent edition. The overall impression is excellent—a thoroughly recommendable purchase.

G. W. MADARAS

Dictionary of Textile Terms

German-English and English-German

Compiled by M. Polanyi. Pp. x + 328. London and New York: Pergamon Press. 1956. Price, 60s. 0d.

Mrs. Polanyi in her preface disarms criticism by

saying: "Suggestions for correcting and amplifying the dictionary will be welcomed".

This makes it easier for the reviewer, because there immediately comes to mind a suggestion that when a word is in common use its literal translation as well as the technical meaning should be given. For instance, *Kunstgriff* is a perfectly respectable German word meaning "ingenious device". It is hardly fair to translate it simply by "artifice" or "trick", both of which words have in English a sinister implication, especially as *Kunstgriff* conveys the idea of a master stroke rather than a trick, which word most Germans would translate by *Trug*. The literal German meaning of the English verb "cure" is *heilen* or *bewahren*. If the curing is one of dehydration, then it is better to translate it by *entwässern* or *trocknen*, but there are curing processes which are not merely drying.

It is right to give the gender of German nouns: English people never remember it, and Germans never forget it. But there is one word, and I believe only one word, namely *Dotter* (egg-yolk), where even the Germans seem to be in doubt. Mrs. Polanyi says that it is masculine, while other German dictionaries say that it is neuter, and one reputable German dictionary gives all three genders—masculine, feminine, and neuter.

The sins of omission are pardonable, because many textile terms have their origin in dialect. They vary from district to district and from factory to factory, and they seldom find their way into print. All the same, a German visiting an English factory may hear the word "hask" used in the sense of harshness of handle, and he would have to consult a pretty comprehensive dictionary to find its meaning. Further, I think that such well known terms as "jean", "lawn", and "Bedford cord" should find a place in a textile dictionary.

Mrs. Polanyi has essayed a task well worth doing, and worth doing well. The printing and the lay-out of the book are excellent.

R. GAUNT

The Textile Recorder Annual Book of the Year 1956

Harlequin Press (1955) Ltd. Pp. 208. Manchester and London. Price, 30s. 0d.

This volume, which is really a survey of the year 1955, is divided into sections dealing with—surveys of trade and industry, technical progress in the mills, new machines and equipment, trends in textile research, the raw-materials position, and textile statistics. Eight pages of "highlights of the year in pictures" may be of greater interest to those photographed than they would care to admit,

and forty pages are devoted to advertisements. Two articles of special interest to members of the Society are *Dyeing for the Knitted-goods Trade* by A. W. Carpenter, which refers especially to the dyeing of Orlon, Acrilan, crimp nylon yarn, and Terylene, and *Dyeing, Printing and Finishing Machinery*.

C.J.W.H.

Bibliographical Abstracts on Redeposition of Soil on Cotton Fabric

Special Technical Publication No. 173

Prepared by ASTM Committee D-12, Subcommittee T-5, Task Committee on Redeposition Methods. Pp. ii + 30. Philadelphia: American Society for Testing Materials. 1956. Price, \$1.00.

A task committee was appointed in 1953 to develop a method of measurement of the redeposition of soil on fabric from aqueous detergent systems (laundering). Thus the 171 abstracts, covering the years 1909-1953, tend to be biased toward indicating contents of original papers that are relevant to testing methods and theories. The bibliography itself is preceded by a review of the literature on the theory and practice of redeposition methods which was presented at a meeting of ASTM Committee D-12 on Soaps and Other Detergents held in March 1955. Subject and author indexes are included.

C.J.W.H.

Natural Rubber Latex and its Applications

No. 5—The Applications of Latex to Textile and Allied Materials

By C. M. Blow. Pp. 135. London: Natural Rubber Development Board. 1956. Gratis.

This handbook is written from the practical standpoint for users and potential users of latex. After an introductory survey, ten chapters are devoted to—general principles; treatment of yarns, etc.; proofing, coating, and impregnation of fabrics; adhesion of rubber to textiles; rubber-textile composite products, and rubber as a finishing agent; treatment of pile fabrics; latex-bonded fibrous structures; manufacture of artificial leather; applications to paper; and miscellaneous applications to textiles, including the use of latex to attach other substances to textiles and as a resist to produce novelty effects. Finally, abstracts are given of the 236 British patent specifications referred to in the text, followed by three appendixes dealing with design of equipment, estimation of rubber content, and qualitative assessment of the distribution of rubber on a textile.

C.J.W.H.

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes. Any publication abstracted may be referred to by members of the Society on application to Dr. C. B. Stevens, Dyeing Department, Leeds University

Badische Anilin- und Soda-Fabrik

PRODUCTS FOR THE TEXTILE INDUSTRY: IV—DYEING AND FINISHING OF RAYON (Viscose, Cuprammonium, and

CELLULOSE ACETATE)—Products suitable for pretreating, bleaching, dyeing, and finishing these fibres are listed, together with brief notes on applications of some of them.

PRODUCTS FOR THE TEXTILE INDUSTRY: VIII—DYEING AND FINISHING OF JUTE AND COIR—A list of auxiliary products for use on these fibres with brief notes on their applications.

BASOLAN FAST DYES—This card contains dyeings in three depths on wool yarn and spun Perlon yarn of five new homogeneous Basolan Fast dyes, viz. Yellow GGL and GRL, Orange GL and RRL, and Red GL. They are designed for dyeing wool, polyamide fibres, and silk in all forms. A feature is that they may be applied to wool from either a strongly acid or a weakly acid dyebath. For wool which is likely to be difficult to dye evenly and for polyamide fibres the weakly acid method is recommended. Dyeings of standard depth on wool and Perlon are rated 5-7 to light and 4 or better to washing (a).

VIALON FAST VIOLET RR—This metal-complex dye is a further addition to the special range developed for dyeing polyamide fibres. It gives bright reddish violets of very good fastness to light and wet treatments and is particularly recommended for use alone and in mixtures on Perlon slubbing. Fastness figures on Perlon include—Light 7, washing (b) 4-5, alkaline milling (b) 4-5.

Cassella Farbwerke Mainkur AG.

INDANTHREN YELLOW F2GC—This vat dye gives bright golden yellows of very good fastness to light and wet treatments on cellulosic fibres. It does not promote photochemical degradation of the fibre or of dyes used in mixtures with it, and it is thus suitable for curtains and furnishings as well as coloured bleach styles. On silk it is of interest for dress cloth but not for materials to be exposed to light for prolonged periods. It has very good levelling properties. Fastness figures on cotton include—Light 6-7, chlorine 4-5, soda boiling 4-5.

Farbenfabriken Bayer AG.

PHTHALOGEN BRILLIANT BLUE IF2G—This phthalocyanine derivative gives very bright blues on cellulosic fibres. It is primarily intended for printing cotton and cuprammonium rayon; for viscose rayon the IF3G brand is more suitable; on cotton only it is suitable for pad-dyeing, particularly for pale colours. Unlike Phthalogen Brilliant Blue IF3G, the IF2G brand already contains the copper compound necessary for development of the final complex. It gives a higher colour yield and more stable printing pastes; it can be printed together with vat, solubilised vat, Rapidogen, and Acramin dyes and aniline black, and is of interest for extremely bright resists under aniline black and Variamine Blue. The Indanthren standard of fastness is claimed without limitation of depth on curtains, furnishings, awnings, and all materials to be repeatedly laundered. Fastness figures on cotton include—Light 7-8, soda boiling 5, chlorine 3-4.

RAPIDOGEN BROWN N-IBRR 54—This stabilised azoic dye gives full reddish browns of very good fastness to light when printed on cellulosic fibres, and can be developed in either neutral or acid steam or by the wet developing process, no addition of Rapidogen Salt A or Developer N being necessary. Prints attaining an agreed minimum depth are claimed to reach the Indanthren standard of fastness for washing styles such as curtains and furnishings. Fastness figures on cotton include—Light 6, washing (c) 4-5, soda boiling (b) 4, chlorine (b) 4.

Farbwerke Hoechst AG.

BLEACHING AUXILIARY HV—This product is designed to suppress the generation of chlorine dioxide in sodium chloride bleach liquors. Addition of 0.5-2 g./litre almost completely inhibits the generation of chlorine dioxide from solutions within the pH and temperature ranges normally employed, and corrosion of metal and risk of injury to operatives is eliminated, the consumption of chlorine being reduced at the same time.

IMPERON DYES FOR PRINTING AND DYEING—This booklet contains prints on mercerised cotton, viscose rayon crêpe, and Perlon of twelve pigments resin-bonded to the fibre by means of oil-in-water emulsions containing

synthetic resin binder in ammoniacal solution (Imperon Binder FA), a heavy aromatic-free hydrocarbon (b.p. 190-230°C.), a fixing agent to promote cross-linking of the binder, water, and the pigment paste. The printed goods are dried at as high a temperature as possible, and pigment fixation is effected by neutral or acid steaming for 5-7 min. On synthetic fibres dry heating for 3-5 min. at < 140°C. is suitable. These dyes may be used alongside vats, solubilised vats, and azoic combinations and under aniline black. When Imperon dyes only have been used, no washing-off is necessary after printing. Advantages claimed for the Imperon process include ease of preparation of very stable print pastes, no need for acids or catalysts which might promote tendering of the material, and good wet fastness of the finished prints, which also show good resistance to sticking and marking off, since the cured resin film has only very low thermoplasticity. Very level pad dyeings of good wet fastness can be obtained using simple aqueous mixtures of pigment and binder. Machine prints on cotton fixed by steaming for 7 min. have been given the following average fastness figures (reference type depth)—Light 7 or better (exception: Red GR), washing (95°C.) 4-5 or better, chlorine 4-5 or better.

Geigy Co. Ltd.

IRGALAN NAVY BLUE 5RL—This neutral-dyeing metal-complex dye gives reddish navies of very good fastness to light and wet treatments on wool and silk, and in combination with other Irgalan and Irganol 8 dyes a full range of navies can be obtained on loose wool, slubbing, yarn, and cloth. On polyamide fibres there is a tendency for bronzing to occur, particularly in full depths, and it is of less value in this field. Fastness figures on wool include—Light 7, washing (b) 4-5, heavy milling 5.

IRGANOL NAVY BLUE 2RLS—This neutral-dyeing acid dye is recommended for navy blues on wool and silk. The dyeings obtained are greener and purer than those using Irgalan Navy Blue 5RL and show less tendency to redden in artificial light. The fastness properties of the dyeings are identical, except that Irganol Navy Blue 2RLS gives somewhat better fastness to acid chlorination. Their dyeing characteristics also are very similar, but the Irganol Navy is not suitable for shading at the boil. Its tendency to bronze on polyamide fibres, particularly when used in full depth, makes it less suitable for dyeing this fibre. Fastness figures on wool include—Light 7, washing (b) 4-5, heavy milling 5.

E. I. du Pont de Nemours & Co. Inc.

DYEING AND FINISHING ORLON TYPE 42 ACRYLIC FIBRE: BULLETIN OR-51—This pamphlet gives details of suitable dyes and dyeing methods for dyeing Orlon and mixtures of Orlon and wool, cellulosic fibres, polyamide fibres, and polyester fibres. Notes on scouring, stripping, printing, bleaching, and finishing are also included, and recommendations are made for dyeing particular forms of material.

DYEING OF BLENDS OF ORLON TYPE 42 ACRYLIC FIBRE AND WOOL—This leaflet describes the production of solid and multicoloured dyeings with neutral-dyeing acid or metal-complex dyes or chrome dyes for the wool and basic dyes for the Orlon. By selecting suitable combinations of dyes and careful control of dyeing conditions both fibres may be dyed simultaneously.

DYEING ORLON ACRYLIC FIBRE AND BLENDS WITH OTHER FIBRES—This is a reprint of a paper by J. F. Laucius, R. A. Clarke, and J. A. Brooks (*Amer. Dyestuff Rep.*, 44, P 362 (1955)).

Sandoz Products Ltd.

METOMEGA CHROME YELLOW GL—This chrome dye is particularly recommended, alone and in mixtures, for dyeing wool by the single-bath method to give yellows of very good fastness to light and wet treatments. It is also of interest for dyeing wool-nylon mixtures and for Vigoureux (mélange) printing. Fastness figures for a metachrome dyeing on wool include—Light 6, alkaline milling 4-5, potting 3.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index

I—PLANT; MACHINERY; BUILDINGS

Flammable Liquids in the Laboratory

S. M. MacCutcheon

Ind. Eng. Chem., 48, 63 A-64 A (Aug. 1956)

Suggested rules and standards are described for the safe storage and handling of flammable liquids in the laboratory. These cover quantities, containers, transfer, disposal, sources of ignition, and general precautions.

W.K.R.

Heat Economy in Textile Processing

W. Simon

Melliand Textilber., 37, 111-115 (Jan. 1956)

PATENTS

Mixing Liquids together or with Solids

Bradford Dyers' Assocn.

BP 754,663

A vertical hollow rotor is partly immersed in the liquid. The rotor is surrounded by a casing which divides the liquid into a relatively static main portion and a moving central portion. The latter is urged upwards by centrifugal force and on arriving at the top of the rotor is thrown outwards on to the surface of the main portion of the liquid.

C.O.C.

Preventing Corrosion of Apparatus by Acid Solution of Chlorites or Chlorine Dioxide

Deutsche Gold- und Silber-Scheideanstalt vormals Roessler

BP 755,196

When bleaching with acid solutions of chlorites or chlorine dioxide, corrosion of the apparatus is avoided if the surfaces with which the liquor is in contact are formed of titanium or an alloy containing Ti as its predominant component.

C.O.C.

Treating Thread

Courtaulds

BP 754,704

A series of rollers, preferably cantilever rollers, are placed in endwise stepped relation and overlapping with the axis of each roller at an angle to the next roller, so that each pair of adjacent rollers forms a thread-advancing device and each roller other than the end rollers of the series forms part of two thread-advancing devices. Arrangements are made for the thread to be wet-processed and dried while passing along the rollers.

C.O.C.

Applying Liquid to Thread advancing in Helical Turns over a Pair of Rollers

Courtaulds

BP 756,308

Continuous Saponification of Cellulose Acetate Filaments

Celanese Corp. of America

BP 755,865

Shrinkable Wrappers for Annular Thread Packages

American Viscose Corp.

BP 755,976

Wet Processing of Textiles

H. Krantz

BP 755,877

In a machine in which the liquor flow is automatically reversible, formation of steam bubbles and locks in the active current of the liquid are avoided if the liquor supply and the discharge pipe of the expansion chamber, placed at the top of the circuit, are separated from each other, so that the liquor is fed first to the heating device and at least some of it fed to the steam pressure space before it can pass to the discharge side.

C.O.C.

Drive for Jig Dyeing Machine

Allmänna Svenska Elektriska (ASEA)

BP 756,530

Continuous Wet Processing of Fabrics

Rodney Hunt Machine Co.

BP 756,708

When fabric is being processed in machines of the types described in BP 664,511 and USP 2,618,142 overdriving the upper rolls enables the fabric to be moved through the liquor at high speeds while it is subjected to tensions much less than those produced in any hitherto available machine.

C.O.C.

Apparatus for Treating Webs by More than One Operation

Manufacture de Machines Modernes

BP 755,281

Means to ensure that in apparatus in which more than

one operation is performed, e.g. printing and embossing, one part of the apparatus can be stopped and re-started only in its proper relationship to the other parts.

C.O.C.

Expander Rolls

J. D. Robertson

BP 754,967

BP 756,136

Silk-screen Printing Machine

L. G. Dubuit

BP 756,133

Apparatus for silk-screen printing on large cylinders.

C.O.C.

Drum Washing, Dyeing, or Dry-cleaning Machine

Isaac Brathwaite & Son Engineers

BP 756,467

Doctors for Paper, etc. Machines

H. E. B. Scott

BP 752,098

The doctor blade is seated in an open-topped slot of the doctor holder and is maintained in position by a thin pressure plate placed at an angle of 5° above, and bearing against, the upper surface of the blade along the whole, or most, of its length. The smooth, streamlined form of the doctor allows the pulp or paper web to travel freely along the upper surface of the pressure plate, along and over the holder.

K.W.

Rope Transfer Means for Webs of Paper, etc.

Lukens Steel Co.

BP 753,958

The paper web is led from one dryer section to another by pairs of rope carriers associated with each section which are transferred above the last roll of the one dryer section and below the first roll of the second dryer section, each of the rolls having take-off and lead-on pulleys, one of which is so placed as to change the direction of travel of the rope passing over it, whereby the tail of the web is gripped between pairs of ropes. This allows the spacing between dryer rolls to be substantially the same throughout the entire dryer.

K.W.

Garment Press

David A. Freeman Corp.

BP 755,684

Preparation and Properties of Glass "Semi-silk" (VI p. 550)

Special Problems in Vat Printing Practice (IX p. 552)

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Fifty Years of Hydrosulphite and Rongalite

A. Janson

Melliand Textilber., 37, 98-100 (Jan. 1956)

The history and development of hydrosulphite and Rongalite since they were first marketed in 1906 are described. All vat dyeing and printing reducing agents used hitherto may be regarded as derivatives of sulphoxyllic acid.

H.K.

PATENTS

Detergent

G. L. Busch

BP 754,705

Adding a polycarboxylated amine to a mixture of a synthetic detergent and a higher aliphatic amide or hydroxyalkylamide results in better foam stability and dirt removal and reduces reprecipitation of the dirt.

C.O.C.

Carboxyalkylcellulose Detergent Composition

Handelsvereeniging Holland

BP 754,714

A mixture of a detergent and a carboxyalkylcellulose has the best washing properties if the cellulosic product is an alkali-metal or ammonium salt of a carboxyalkylcellulose which has been produced by etherifying cellulose in an inert organic solvent. The carboxyalkylcellulose should have a degree of substitution of 0.4-0.6 and a fibre content of > 4%, and its 1% aq. soln. containing 2.5% by wt. of Na₂CO₃ and 2.5% of NaHCO₃ a viscosity of 10-25 centipoises at 20°C. measured in a Höppler viscometer.

C.O.C.

Polyoxyalkylated *tert.*-Carbinamines

Rohm & Haas Co.

BP 754,670

Compounds of formula—

(m = 3–100; n = 2 or 3; R¹, R², and R³ = Alk of together 7–23 C, e.g.—

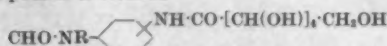
(CH₃)₃C-CH₂-C(CH₃)₂-NH-[CH₂-CH₂-O]_{3–100}-CH₂-CH₂-OH are useful as wetting agents, emulsifiers, corrosion inhibitors, and bactericides in organic solvents and/or aqueous systems. C.O.C.

Substituted Phenylenediamines—Solvents for Sulphuric Esters of Leuco Vat Dyes

General Aniline

USP 2,721,211

Compounds of formula—



(R = Alk or hydroxyalkyl; the acylated N atom is either *meta* or *para* to the other N atom), e.g. *N*-formyl-*N*-methyl-*N'*-gluconyl-*p*-phenylenediamine, are good solubilising agents for ionic compounds in polar solvents and especially for alkali-metal salts of sulphuric esters of leuco vat dyes in water. They are prepared by reaction of the amino-*N*-alkyl(or *N*-hydroxyalkyl)formanilide with gluconic lactone to form the corresponding gluconyl derivative. C.O.C.

Polymeric Zirconium Compounds having Surface-active Properties in Hydrocarbon Solvents—Water-repellent Agents

DuP

BP 755,558

The products obtained by reaction of a compound of formula Zr(OR)₄ (R = Alk, alkoxyalkyl, cycloalkyl, Ar, or aralkyl) with an anhydrous aliphatic carboxylic acid in presence or absence of an organic solvent are readily soluble in organic solvents to yield solutions having surface-active properties. They may also be used for imparting water-repellency to textiles. Thus cotton sateen is rendered water-repellent by padding it with a 5% soln. of polymeric ethoxyzirconium linseed-oil acylate in xylene and drying in air. C.O.C.

Improving the Efficiency of Carrotting Agents

Deutsche Gold- und Silber-scheideanstalt vormals, Roessler

BP 757,175

The efficiency of carrotting agents such as H₂O₂ or per-salts is much improved by adding formamide, in many cases enabling the addition of acid to be avoided. C.O.C.

Atmospheric Oxidation of Alkali Sulphide Solutions and its Effect on the Unhairing and Dewooling of Skins (XII p. 554)**IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS****Fluorescence of Dyes in Solution**

H. E. Millson

Amer. Dyestuff Rep., 45, 434–440, 459 (2 July 1956)

The fluorescence characteristics of 46 dyes are tabulated. J.W.B.

Mixed-acid Nitration of Toluene

H. M. Brennecke and K. A. Kobe

Ind. Eng. Chem., 48, 1298–1304 (Aug. 1956)**Toluene Nitration Kinetics**

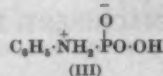
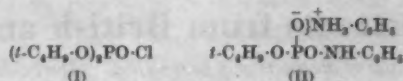
A. J. Barduhn and K. A. Kobe

Ind. Eng. Chem., 48, 1305–1315 (Aug. 1956)**New Method of Phosphorylating Aromatic Amines**

H. Goldwhite and B. C. Saunders

Chem. and Ind., 663–664 (7 July 1956)

A new method of phosphorylating amines depends on the ready thermal dealkylation of *tert.*-butyl phosphoric esters. Thus, the product (presumed to be di-*tert.*-butyl phosphorochloridate (I)) from di-*tert.*-butyl phosphite and *N*-chlorosuccinimide reacts with aniline to yield the anilinium salt of mono-*tert.*-butyl *N*-phenylphosphoramidate (II). This salt gives a quantitative yield of *N*-phenylphosphoramidic acid (the required *N*-phosphorylated amine (III)) when maintained at 80°C. and 1 mm. for 15 hr. Similar compounds have been prepared from *p*-chloro-, *p*-bromo-, and *p*-methyl-aniline.



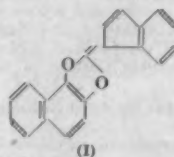
A.J.

Thermal Decomposition of Naphthalene 1:2- and 2:1-Diazo-oxides

P. Yates and E. Robb

Chem. and Ind., 794–795 (4 Aug. 1956)

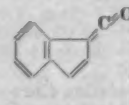
Bamberger's bis-naphthalene oxide C₂₀H₁₂O₂ obtained by decomp. of either naphthalene 1:2- or 2:1-diazo-oxide in boiling xylene, is now shown to have formula I. This can be rationalised in terms of loss of nitrogen from the diazo-oxide to afford an intermediate (II), which rearranges to the keten III, and this reacts with unrearranged II; such a pathway accommodates Bamberger's observation, now confirmed, that the same product may be obtained from either diazo-oxide.



(I)



(II)



(III)

H.H.H.

Triphenylmethane Dyes

L. Roessens

Compt. rend. 27^e Congr. intern. Chim. ind., Brussels, (3) (1954):*Industrie chim. belge*, 20, Spec. No., 641–645 (1955):*Chem. Abs.*, 50, 9909 (25 July 1956)

An account of the preparation of derivatives of 4:4'-dimethoxydiphenylmethane in which the central C atom bore one of the following heterocyclic substituents—radicals of pyridine, quinoline, 2-methylindole, carbazole, benzothiazole, benzoselenazole, phenothiazine, phenoxazine, phenoselenazine, and sulphondiphenylamine. The results of spectrophotometric absorption observations for acid solutions of the dyes are discussed in terms of the theories of Lewis and Calvin, Pauling, Brand, and Brunings and Corwin. 26 references. C.O.C.

Complex Compounds of Polymethine Dyes with Silver Ions. I.—Formation of Silver Ions of Carbo- and Polycarbo-cyanines

K. I. Pokrovskaya, I. I. Levkoev, and S. V. Natanson

Zhur. fiz. khim., 30, 161–171 (1956):*Chem. Abs.*, 50, 9925 (25 July 1956)

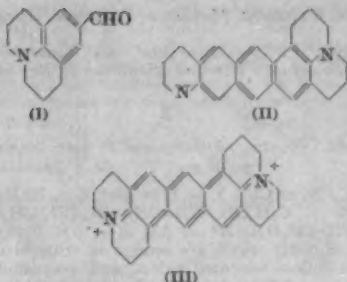
The ability of 32 symmetrical cyanine dyes containing different heterocyclic residues and polymethine chains of various lengths to form complexes with Ag ions is greater the greater the basicity of the dye. C.O.C.

Coloured Salts derived from certain Anthracenes

F. H. C. Stewart

Chem. and Ind., 579 (30 June 1956)

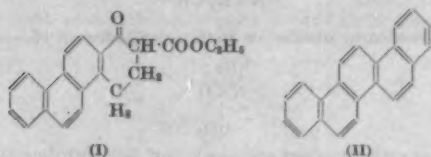
9-Formyljulolidine (I) when treated with tin and dil. HCl gave a 70–75% yield of the new anthracene derivative II. II with dil. HCl and H₂O₂ gave a deep red soln., reasonably stable to heating, from which a blue chloride may be crystallised as a hydrate which in turn gives a picrate, chloroplatinate, and perchlorate. Analysis shows these to be salts of the quinonoid di-imonium cation (III). 2:6-Bisdimethylaminoanthracene (IV), prepared as for II from *p*-dimethylaminobenzaldehyde (Albano, *As. Assoc. quim. argent.*, 34, 18 (1946)), when treated with acidic H₂O₂ gives a much less intense red soln., which rapidly becomes colourless on heating. The enhanced stability of III is considered to be due to permanent coplanarity, which is not possible with the analogous cation from IV. 2:6-Bisdiethylaminoanthracene gives only a very faint colour with acidic H₂O₂, owing to weak conjugation caused by steric hindrance of the diethyl-amino group.

**New Synthesis of Picene**

D. Nasipuri

Chem. and Ind., 795 (4 Aug. 1956)

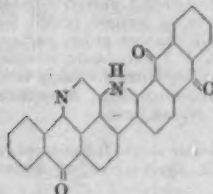
Ethyl 4:6-dioxoheptane-1:5-dicarboxylate is alkylated with β -1-naphthylethyl bromide in presence of ethanolic sodium ethoxide, and the product cyclised with conc. H_2SO_4 to afford γ -(2-carboxy-3:4-dihydro-1-phenanthryl)-butyric acid, whose diethyl ester on dehydrogenation with the calc. amount of 8 and subsequent hydrolysis gives the corresponding aromatic acid, whose diethyl ester undergoes a Dieckmann cyclisation to 2-ethoxycarbonyl-1-oxo-1:2:3:4-tetrahydrochrysene (I). I reacts with a methanolic soln. of the methiodide of 1-N-piperidino-3-butanone to give 2-ethoxycarbonyl-2- γ -oxobutyl-1-oxo-1:2:3:4-tetrahydrochrysene, which acid cyclisation converts to an unsaturated ketone. The latter reduces with $LiAlH_4$ to an alcohol, which is converted into picene (II) by heating to 340°C. in presence of 30% Pd-C catalyst.

**Nitrogen Analogue of Indanthren Olive Green B from Benzoyl-3-azabenzanthrone**

M. Yokote and S. Kobayashi

J. Chem. Soc. Japan, Ind. Chem. Sectn., 58, 677-678 (1955); *Chem. Abs.*, 50, 10411 (25 July 1956)

3-Bromo-1-azabenzanthrone (0.25 g.), 1-aminoanthraquinone (0.134), Na acetate (0.24), $CuCl_2$ (0.035), and nitrobenzene (5.25) were refluxed on an oil-bath for 12 hr., washed, extracted with hot alcohol and hot glacial acetic acid, and boiled with acid water, and the residue was boiled with *o*-dichlorobenzene. The precipitate (0.144) was fused with KOH (8), phenol (6.4), and alcohol (1.6) at 135-145°C. for 2 hr., boiled with water (100 c.c.), acidified, and filtered to yield a green dye (0.142). Purifying with chlorobenzene gave a greenish-blue dye (0.634), which, because of the similarity of its absorption spectra and n -values to those of Indanthren Olive Green B, appears to have the structure—



C.O.C.

Spectroscopic Studies on Dyes. II—Structure of NN'-Dimethylindigo

J. Weinstein and G. M. Wyman

J. Amer. Chem. Soc., 78, 4007 (20 Aug. 1956)

Unlike other indigoid dyes, the green NN'-dimethylindigo is very soluble in organic solvents and has little affinity for cellulose. The wavelength of its visible absorption band is very dependent on the nature of the solvent,

hydroxylic solvents, in particular, causing a strong bathochromic shift. The absorption band shifts to still longer wavelengths with the solid dye. Irradiation of a solution of the dye with yellow or red light causes rapid *trans* \rightarrow *cis* isomerisation; in the dark this reaction reverses almost instantaneously. This dye also forms hydrogen-bonded complexes with phenol and ethanol. It seems that, probably because of the steric effect of the methyl groups, the central double bond of this dye has much more of a single-bond character than in other indigoid or thioindigoid dyes. C.O.C.

Vivid Light-fast Organic Pigments

V. C. Vose

Official Digest Federation of Paint & Varnish Production Clubs, 28, (377) Part 2, 1-48 (June 1956)

The following properties of 36 organic pigments of the phthalocyanine, anthraquinone vat dye, thioindigoid vat dye, and azo types are given—Munsell measurements of hue, value, and chroma of each pigment with TiO_2 in an alkyl enamel film; similar data for the film after a 1-year exposure at Miami (Flor.); the spectrophotometric curves from which these figures were derived; and colour chips printed by the McCorquodale process from lacquers coloured with the pigments themselves at approximately the same colour concentrations as the panels used in the exposures. R.K.F.

Properties and Composition of the Bile Pigment giving a Direct Diazo Reaction

E. Talafant

Nature, 178, 312 (11 Aug. 1956)**Constitution of Cernuoside, the Yellow Pigment of the Flowers of *Oxalis cernua***

A. Ballico and G. B. Marini-Bettolo

Gazz. chim. ital., 85, 1319-1328 (1955); *Chem. Abs.*, 50, 10073 (25 July 1956)

Proof that this pigment is the 4-glucoside of aureusidin. C.O.C.

Swerchirin, a New Xanthone from *Swertia chirata*

S. R. Dalal and R. C. Shah

Chem. and Ind., 664 (7 July 1956)

Acidification of the alkaline extract from the ether extractive of the powdered stems of *Swertia chirata* yields dark yellow needles (m.p. 185-186°C.), named *swerchirin* (I). I contains two $O-CH_3$ and two OH groups and on methylation yields 1:3:5:8-tetramethylxanthone, whilst demethylation gives 1:3:5:8-tetrahydroxyxanthone (II). I is therefore a dimethyl ether of II. A.J.

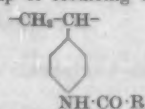
PATENTS

Colour Couplers

Kodak

BP 754,628

Polymeric couplers containing no alkali-solubilising groups and made up of recurring units of formula—



(R = an organic radical having coupling properties) together with a water-insoluble organic crystalloidal compound of b.p. > 175°C. which is a solvent for both the couplers and the dye formed from them are dispersed in a photographic emulsion. The coupler has little tendency to diffuse into or from the emulsion during storage or processing. C.O.C.

Colour Couplers

Kodak

BP 754,306

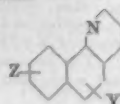
Amides and esters of salicylic acid having an alkoxy, amino, or acylamino substituent in the nuclear position *para* to the amide or ester group have much better coupling rates in colour-development processes and yield much more stable dye images than do either salicylic acid or unsubstituted salicylamides. C.O.C.

Cyan Azine Colour Formers

General Aniline

BP 757,045

Compounds of formula—



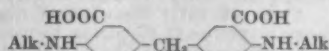
(Z = H, OH, NH₂, aliphatic amino, aromatic amino, acylamino, heterocyclic amino, aromatic carbonamido, or aliphatic or aromatic sulphonylamino; Y = OH, NH₂, or NH-SO₂-R; R = aliphatic or aromatic radical, e.g. benzo(h)quinolin-5-yl or 9-methylsulphonylamino-6-amino-benzo(h)quinoline, have good coupling speed and yield brilliant dye images. C.O.C.

Diazoamino Compounds for Azoic Printing Compositions

Fran

BP 753,591

The diazo compounds from amines of the type generally used in the production of azoic dyes and prints are condensed with compounds—



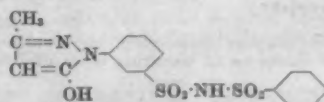
(Alk may carry substituents such as OH, O-CH₃, COOH, SO₃H, OPO₃H₂, CO-N<, etc.) to give diazoamino compounds which readily regenerate the diazo compound. With diazo compounds derived from amines of a more basic character than 2:5-dichloroaniline, neutral steaming will regenerate the diazo compound; whereas with diazo compounds from amines of a less basic character, acid steaming may be required to ensure rapid regeneration. Thus *N*-methylantranilic acid is treated with formaldehyde in presence of aq. HCl for 1 hr. cold and 2 hr. at the boil to give 4:4'-di-*N*-methylanilino-diphenylmethane-3:3'-dicarboxylic acid. This is condensed with a diazo soln. prepared from 2-amino-5-chlorotoluene, to give the diazoamino compound, which is mixed with sodium 3-hydroxy-2-naphthoanilide and anhydrous sodium sulphate. After suitably thickening, printing on cotton fabric, and steaming for 5-6 min. in neutral steam, bright yellowish-red prints are produced. E.S.

Preparation of 5-Pyrazolone Derivatives

Ciba

BP 754,660

Hydrazines of general formula H₂N-NH-R-SO₂-NHX (R = aryl of benzene series; X = the acyl radical of an organic sulphonic acid, e.g. ethanesulphonyl or benzenesulphonyl) are condensed with diketene or alkyl acetoacetates to give derivatives of 3-methyl-1-phenyl-5-pyrazolones. Thus aniline-*m*-sulphon-*N*-(benzenesulphonyl)amide is diazotised, and the diazo soln. treated with aq. sodium bisulphite and NaOH to give the hydrazine. Condensation with ethyl acetoacetate then gives the pyrazolone—



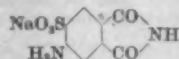
E.S.

4-Aminophthalimide-5-sulphonic Acid

FBY

BP 751,570

4-Aminophthalimide is sulphonated by treating with chlorosulphonic acid in nitrobenzene at 120-140°C. The precipitated internal salt is filtered off, and may be converted to the Na salt by neutralising with aq. Na₂CO₃ and adding NaCl. The resulting—



is an intermediate for preparing water-soluble phthalocyanines. R.K.F.

Azo Derivatives of β-Naphthylamine prepared indirectly from its 1-Sulphonic Acid

Ciba

BP 756,148

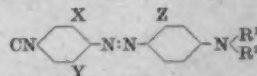
2-Naphthylamine-1-sulphonic acid (I) couples with diazo compounds in the 1-position with removal of the sulpho group, so that use of the carcinogenic β-naphthylamine may be avoided. In some cases brighter colours are obtained by adopting this indirect method. Thus diazotised 2-amino-6-nitrophenol-4-sulphonic acid is coupled with I in presence of NaOH and naphthalene-α-sulphonic acid at 50°C. The brown-violet monoazo dye so produced is identical with that produced by using β-naphthylamine instead of I. E.S.

Orange to Crimson Monoazo Disperse Dyes

ICI

BP 756,079

Derivatives of *p*-cyananiline are diazotised and coupled with suitable *NN*-dialkylanilines to give monoazo compounds—



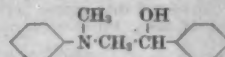
(X = Hal, CN, or CF₃; Y = H or Hal; Z = H, Hal, CH₃, or CF₃; R¹ = C₂H₄OH, CH₂-CN, CH₂-CH₂-CH₂OH, or CH₂-CH(OH)-CH₂OH; R² = Alk of < 5 C, C₂H₄OH, or CH₂-CH₂-CH₂-OH) which are orange to crimson disperse dyes for cellulose acetate, nylon, and polyester fibres. Thus 4-amino-3-chlorobenzonitrile is diazotised and coupled with *NN*-bis-*p*-hydroxyethylaniline. The product dyes cellulose acetate and nylon yellowish red of similar brightness and fastness to light on the two fibres. E.S.

Monoazo Disperse Dyes derived from Condensation Products of Arylamines and Styrene Oxide

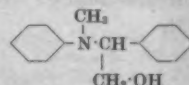
Deutsche Gold- und Silber-Scheideanstalt vormals Roessler

BP 756,112

Equivalent quantities of a primary or a secondary amine of the benzene series and styrene oxide are heated, preferably in an inert solvent in presence of a catalyst, to give products which may be used as coupling components in the preparation of disperse dyes. Thus *N*-methylaniline, styrene oxide, xylene, and a little glacial acetic acid are boiled together for 5 hr. After distilling off the xylene, the principal product (I)—



is separated by distillation from a small amount of—



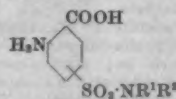
I gives with diazotised aniline a yellow, and with diazotised *p*-nitroaniline a red, disperse dye for cellulose acetate. E.S.

Yellow Chromable and Metal(Chromium)-complex Monoazo Dyes for Wool, Nylon, etc.

S

BP 753,550

Monoazo dyes prepared by diazotising 4- or 5-sulphonamides of anthranilic acid—



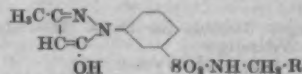
(R¹ = Alk, cycloalkyl, aralkyl, or aryl free from SO₂H and COOH groups; R² = H or Alk, or with N and R¹ forms a heterocyclic residue) and coupling with 1-aryl-3-methyl-5-pyrazolones free from SO₂H, COOH, or sulphonamide groups may be chromed in substances, on the fibre, or in the dyebath. Thus the 5-sulphonmethylamide of anthranilic acid is diazotised and coupled under alkaline conditions with 3-methyl-1-phenyl-5-pyrazolone, and the monoazo compound so formed boiled for 20 hr. with a soln. prepared from chrome alum, tartaric acid, and aq. NaOH, to give a 1:2 chromium complex, which dyes wool and nylon yellow from a weakly acid bath. E.S.

Metal(Chromium)-complex Monoazo Pyrazolone Dyes for Wool, Nylon, etc.

S

BP 754,270

2-Amino-4-chloro(or methyl)-6-nitrophenol is diazotised and coupled with derivatives of 3-methyl-1-phenyl-5-pyrazolone—

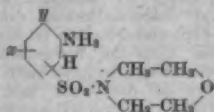


(R = CH₂-OH, CH₂-O-CH₃, CH₂(OH)-CH₂, CH₂-CH₂OH, or CH₂-CH₂-O-CH₃), and the monoazo dyes so formed are converted into their 1:2 chromium complexes, which dye wool and nylon from neutral or weakly acid baths.

Thus 2-amino-4-chloro-6-nitrophenol is diazotised, and coupled with a soln. in aq. NaOH of 3-methyl-1-phenyl-5-pyrazolone-3'-sulphon- β -hydroxyethylamide. The monoazo compound so formed is heated in formamide with ammonium chromium sulphate and sodium acetate, to give the chromium complex, which dyes wool, silk, and nylon bluish red. E.S.

Metal(Cobalt and Chromium)-complex Monoazo Dyes containing a Morpholine Ring

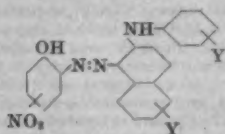
S BP 753,987
Sulphonmorpholides of *o*-aminophenol and its derivatives—



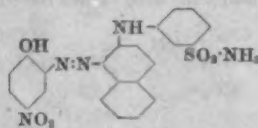
($x = H, \text{Hal}, \text{NO}_2$, or Alk of 1-6 C; $y = OH$ or $O\text{-Alk}$) are diazotised and coupled with suitable *ortho*-coupling phenols, naphthols, etc. free from COOH and SO_3H groups, and the monoazo compounds so formed converted into their 1:2 Co or Cr complexes. The products dye wool, nylon, leather, etc. Thus 2-aminophenol-4-sulphonmorpholide is diazotised and coupled with 1-*m*-chlorophenyl-3-methyl-5-pyrazolone in presence of Na_2CO_3 . The resulting monoazo compound, dissolved in aq. NaOH, is boiled with sodium dichromate in presence of NaOH. The metal complex so formed dyes wool and nylon orange from a neutral or acetic-acid bath. E.S.

Metal(Cobalt)-complex Monoazo Dyes for Wool, Nylon, etc.

Ciba BP 755,113
The metal complexes containing 1 atom of Co to 2 mol. of monoazo dyes free from SO_3H and COOH groups—



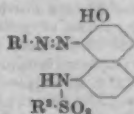
(one Y = sulphonyl or subst. sulphonyl; the other Y = H), or 1 mol. of such a dye and 1 mol. of an *co*'-dihydroxy-monoazo dye free from SO_3H and COOH groups, dye wool, nylon, etc. from neutral or weakly acid baths. Thus 3-hydroxy-2-naphthoic acid is heated in an autoclave with metanilamide to give *N*-*m*-sulphamylphenyl- β -naphthylamine, which is dissolved in dimethylformamide containing 30% aq. HCl, and the hot soln. is run into the diazo compound prepared from 2-amino-4-nitrophenol, giving the monoazo compound—



This is dissolved in aq. NaOH, and stirred with cobalt sulphate at 80°C. to give the metal complex, which dyes wool olive from a neutral or acetic-acid bath. E.S.

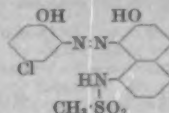
Metallisable Monoazo Dyes for Wool, Nylon, etc.

FBY BP 755,853
Monoazo dyes—



($R^1 =$ aryl of benzene series free from SO_3H and COOH groups but having a group capable of metal-complex formation *ortho* to the azo link; $R^2 =$ Alk or subst. Alk) may be metallised in substance or in the dye bath and give dyeings on wool, nylon, etc. of superior fastness to milling

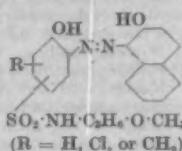
and acid, and in some cases to light, than the similar dyes of BP 684,646 (cf. J.S.D.C., 69, 95 (1953)), which have an alkylcarbonylamino group in place of the alkylsulphonylamino group. Thus 1-amino-7-naphthol is treated with methanesulphonyl chloride, and the *N*-methylsulphonyl compound so formed is coupled in presence of Na_2CO_3 with diazotised 2-amino-4-chlorophenol to give—



which dyes wool blue-grey by the metachrome process. Its Cr complex dyes wool grey-blue, and its Co complex reddish navy blue, from neutral or weakly acid baths. E.S.

Metal(Chromium and Cobalt)-complex Monoazo Dyes for Wool, Nylon, etc.

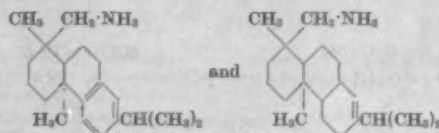
S BP 755,576
Metal complexes containing 1 atom of Cr or Co to 2 mol. of monoazo compounds—



dye wool, nylon, etc. bordeaux and violets from neutral baths. Thus diazotised 2-aminophenol-4-sulphonyl-methoxypropylamide is coupled with β -naphthol in presence of NaOH and Na_2CO_3 . The monoazo compound so formed is treated at 80-85°C. with CoSO_4 in presence of NaOH to give the cobalt complex, which dyes wool, leather, silk, and nylon bordeaux. E.S.

Yellow Oil- and Spirit-soluble Monoazo Dye

American Cyanamid Co. USP 2,721,111
The salts formed by combining the monoazo dye aniline \rightarrow 1-(2-chloro-5-sulphophenyl)-3-methyl-5-pyrazolone with aminoalkyl-polyalkyl-polyhydrophenanthrenes—



have much higher solubility in alcohols, hydrocarbons, and chlorinated hydrocarbons than the salts from the same dye and cyclohexylamine and dicyclohexylamine. E.S.

Orange Metal-complex Disazo Dyes for Leather

BASE BP 753,771

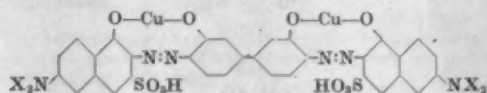
The metal (Cu, Cr, Co, Ni, Al, etc.) complexes of disazo dyes formed by coupling tetrazotised 4:4'-diaminodiphenylmethane-3:3'-dicarboxylic acid (I) with two mol. of the same or different coupling components, of which one at least is a pyrazolonesulphonic acid, dye leather yellow-orange. Thus, anthranilic acid is treated with formaldehyde in presence of aq. HCl at 60°C., and the resulting acid soln. of I is tetrazotised by addition of sodium nitrite and coupled with 2 mol. of 3-methyl-1-*m*-sulphophenyl-5-pyrazolone in presence of sodium acetate. The chromium complex of the resulting disazo compound dyes leather of various tannages reddish yellow. E.S.

Metal(Copper and Nickel)-complex Disazo Direct Dyes

Ciba BP 756,190

Tetrazotised benzidine derivatives having metallisable groups in the 3:3' positions (OH , COOH , or $\text{O-CH}_2\text{COOH}$) are coupled with 2 mol. of *ortho*-coupling naphthol-sulphonic acids, at least one of which contains a hydroxy-alkylamino group, the N atom being directly attached to the naphthalene nucleus. The hydroxyalkyl groups in the

resulting disazo compound are converted into sulphate ester groups, and the products are then metallised with Cu or Ni salts to give complexes which dye cellulosic fibres. Thus tetrazotised 3:3'-dihydroxybenzidine is coupled with 2 mol. of *NN*-bis- β -hydroxyethyl-J acid under alkaline conditions, and the disazo compound so formed is treated with a mixture of pyridine and chlorosulphonic acid at 40–55°C. Treatment at 70°C. with ammoniacal CuSO_4 gives the complex—



(X = $\text{CH}_2\text{CH}_2\text{O}\text{SO}_3\text{H}$), which dyes cotton pure blue.

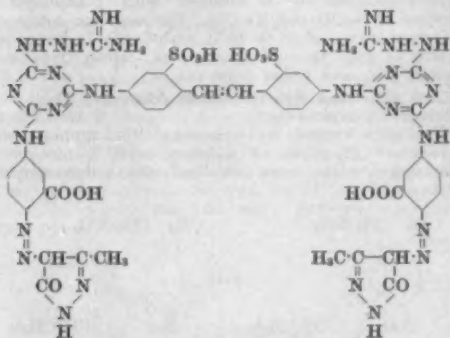
E.S.

Dis- and Poly-azo Direct Dyes for Aftercoppering

General Aniline

BP 751,997

Dyes derived from cyanuric chloride by condensing it with 2 mol. of suitable amino compounds, such as amino-azo dyes or aminostilbenes, and with 1 mol. of guanylhydrazine, semicarbazide, or thiosemicarbazide, give higher wash fastness after treatment on the fibre with a mixture of a copper salt and partially polymerised cyano-guanidine-formaldehyde than do similar dyes in which the third chlorine atom of the cyanuric chloride is condensed with e.g. aniline, *N*-methylaniline, or methylamine. Thus 4:4'-diamino-2:2'-disulphostilbene is condensed with 2 mol. of cyanuric chloride, and the product then condensed with 2 mol. of the aminoazo compound obtained by reducing the NO_2 group of the nitroazo dye 5-nitroanthranilic acid to 3-methyl-5-pyrazolone. Replacement of the third Cl of the cyanuric chloride residues is then effected by treatment with guanylhydrazine in presence of aq. NaOH at 90°C., to give the dye—



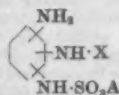
It dyes cotton orange-yellow, fast to light and washing after treatment on the fibre with a mixture of a copper salt and partially polymerised cyano-guanidine-formaldehyde.

E.S.

N-Sulphotriaminobenzenes

Fran BP 753,983

N-Sulphotriaminobenzenes of formula—



(X = H or SO_3A ; A = H or cation) suitable for printing oxidation browns and blacks are made by treating with a sulphonating agent a benzene derivative substituted by NH_2 , by a group convertible to NH_2 by reduction or hydrolysis, and by a group which is either NH_2 or convertible thereto. Thus 1-acetylamino-4-amino-3-nitrobenzene is warmed to 50°C. for 2 hr. with a mixture of chlorosulphonic acid and pyridine, and poured into aq. Na_2CO_3 , the pyridine then being removed by steam-distilling. After separating, the product is first reduced with Fe and acetic acid and then hydrolysed with aq. NaOH.

R.K.F.

Grey Dischargeable Trisazo Direct Dyes

FBY

BP 754,562

Trisazo dyes of the type $\text{A-NH}_2 \rightarrow \text{B-NH}_2 \rightarrow \text{C-NH}_2 \rightarrow \text{D}$ (A = radical of a mono- or di-sulphonic acid of 1-amino-8-naphthol; B and C = radicals of benzene or naphthalene series coupling in *para* position to NH_2 ; D = a mono- or di-sulphonic acid of 2-amino-8-naphthol or 1-amino-7-naphthol coupling in *ortho* position to OH) are dischargeable direct greys of better fastness to light than similar dyes which do not contain sulphonic acids of 1-amino-8-naphthol as initial diazo components. It is preferable to convert these sulphonic acids of 1-amino-8-naphthol into *O*-esters by reaction with benzene(or *p*-toluene)sulphonyl chloride before diazotisation, the ester group being removed from the trisazo dye finally by alkaline hydrolysis. Thus the benzenesulphonyl *O*-ester of H acid is diazotised and coupled with *p*-cresidine, the aminomonazo dye is diazotised and coupled with α -naphthylamine, and the aminodisazo dye so formed is diazotised and coupled with 2-amino-8-naphthol 3:6-disulphonic acid in presence of Na_2CO_3 . Heating with aq. NaOH hydrolyses the ester group and yields the trisazo dye, which gives reddish greys on cellulosic fibres.

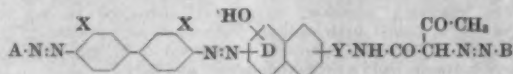
E.S.

Metallisable Trisazo Direct Dyes

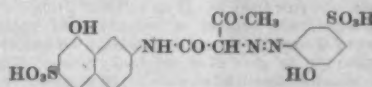
Gy

BP 755,571

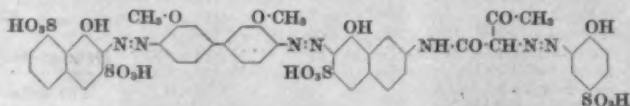
Trisazo dyes of formula—



(A = radical of a naphtholsulphonic acid having its OH group *ortho* to the azo link; X = substituent capable of metal-complex formation; Y = direct link or a bridging group such as $\text{NH-CO-C}_6\text{H}_4$ or a cyanuric chloride residue; B = residue of a diazo compound of the benzene or the naphthalene series having COOH or preferably OH *ortho* to the azo link; in D, OH is *ortho* to the azo link) may be metallised with e.g. Cu or Co salts in substance or on the fibre, or partly in substance and partly on the fibre, giving grey-violets, grey-blues, or grey-greens of good fastness to light, washing, and crease-resist finishes on cotton and viscose rayon. Thus γ acid is treated with diketene to give the *N*-acetoacetyl derivative, which is coupled with diazotised 2-aminophenol-4-sulphonic acid in presence of aq. Na_2CO_3 to give the monoazo dye—



This is coupled with the diazo-monoazo compound formed by coupling tetrazotised dianisidine with 1 mol. of 1-naphthol 3:8-disulphonic acid, to give the trisazo dye—



Heating at 90°C. with ammoniacal copper sulphate gives the copper complex, which dyes cellulose greenish grey.

E.S.

Sulphur Dyes from 4-Hydroxydiphenylamines

Gy

BP 753,764

4-Hydroxydiphenylamines, which may be substituted with Hal and Alk, are fused with iron-free M_2S_x (M = alkali metal; $x = 1.5-3.0$) or a corresponding mixture of M_2S and S in the presence of a water-soluble organic solvent of b.p. > 100°C. to produce red to brown sulphur dyes. Thus 4-hydroxydiphenylamine is heated for 60 hr. at 110°C. with Na_2S and S in ethylene glycol monomethyl ether containing water. The solvent is removed by steam-distilling, air passed through at 40–60°C., and the precipitated dye filtered off. It is purified by stirring with aq. NaOH and aerating.

R.K.F.

Acylaminoanthraquinone Pigments

S

BP 750,969

1:4-Diamino-2-alkyl(or aryl)oxyanthraquinones are

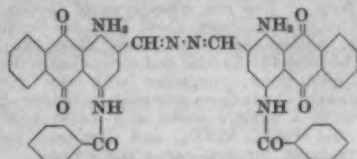
monoacylated with an aromatic carboxylic acid (or chloride) to produce red to violet pigments suitable for printing textiles and colouring paper and plastics. Thus 1:4-diamino-2-ethoxyanthraquinone is heated at 90–95°C. with 4-methylbenzene-1-carboxyl chloride in chlorobenzene containing pyridine. The resulting bisacylamino derivative is treated with H_2SO_4 at room temp. to hydrolyse the 1-acylamino group. R.K.F.

Aminoanthraquinone Azines—Vat Dyes

FBy

BP 750,977

The bisaminoanthraquinonyl azines of BP 148,339 and 150,709 are fast to light and washing if nuclear-substituted with Cl , SO_2-Alk , SO_2-NH_2 , and $CO-NH-Ar$. These substituted derivatives are prepared by treating the corresponding 1-amino-2-methylanthraquinone first with nitrobenzene and alkali to form the azomethine, which is then condensed with hydrazine. Alternatively the 2-aldehyde or 1:2-*iso*-oxazole is treated directly with hydrazine. Thus the dye—



is made by first refluxing 1-amino-4-benzoylamino-2-methylanthraquinone in nitrobenzene with K_2CO_3 . The phenyl-azomethine so formed is filtered off hot and refluxed in acetic acid with hydrazine hydrate.

R.K.F.

Sulphuric Esters of Leuco 1:3:4-Oxadiazole Vat Dyes

ICI

BP 753,494

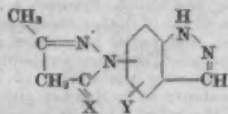
Vat dyes containing one or more 1:3:4-oxadiazole groupings are converted to their leuco sulphuric esters either by treating the dye itself with a sulphating agent in the presence of a metal and a tertiary base or organic amide derived from a secondary amine, or by treating a metal salt of the leuco dye with a sulphating agent in presence of a tertiary base or amide. Thus 2:5-bis-(1-amino-2-anthraquinonyl)-1:3:4-oxadiazole and Cu powder are added at 20°C. to a mixture of chlorosulphonic acid and pyridine. After stirring for 16 hr. the whole is poured into aq. Na_2CO_3 and ice, Na_2S is added, and the copper residues are filtered off. The leuco sulphuric ester is separated from the filtrate by concentrating *in vacuo* and adding KCl. R.K.F.

1-Indazolyl-3-methyl-5-pyrazole Derivatives

ICI

BP 753,573

Intermediates useful for preparing metallisable azo dyes for cotton, having the formula—



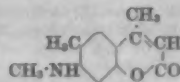
(X = O or NH; Y = H or SO_2H) are made by diazotising the appropriate aminoindazole or its sulphonic acid, reducing to the hydrazine, and then either— (i) condensing with acetoacetic ester or diketene and NH_3 , and cyclising to the pyrazolone; or (ii) condensing with diacetonitrile and cyclising to the aminopyrazole. Thus 6-aminoindazole is diazotised, and aq. $ZnCl_2$ added. The double salt is filtered off, and reduced with $SnCl_2-HCl$ at < 10°C. After filtering off, the product is stirred with acetoacetic ester and aq. NaOH, first at 35–40°C. for 1 hr. and then at 80–85°C. for 4 hr. The resulting compound is separated, after filtering, by adding acetic acid, and purified by dissolving in dil. HCl and precipitating with sodium acetate. R.K.F.

4 : 6 - Dimethyl - 7 - methylaminocoumarin — Fluorescent Brightening Agent for Protein Fibres, Nylon, Cellulose Acetate, etc.

LBH

BP 755,117

The compound—



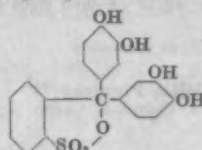
has high-intensity fluorescence at the violet end of the spectrum, it is applicable from acid, neutral, or alkaline baths, and less than 0.01% of it effectively whitens wool. It can be prepared by refluxing an alcoholic solution of *N*-methyl-*m*-aminocresol with anhydrous $ZnCl_2$ and ethyl acetoacetate for 16 hr., after which, on cooling, the zinc chloride compound of 4:6-dimethyl-7-methylaminocoumarin crystallises out. This on heating in water for 30 min. at 70°C. yields the free coumarin derivatives. C.O.C.

Catecholsulphonphthalein

Z. Vodák and O. Leminger

BP 754,367

Pure catecholsulphonphthalein—



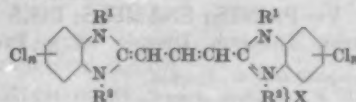
is obtained as crystals by condensing *o*-sulphobenzoic anhydride with catechol at 80–130°C. C.O.C.

Symmetrical Carbocyanine Dyes

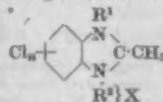
Kodak

BP 754,546

Dyes of formula—



(R^1 = Alk, allyl, or Ar; R^2 = Alk or allyl; X = acid radical; n = 1 or 2), e.g. 5:5'-dichloro-1:1':3:3'-tetraethylbenziminazolocarbocyanine iodide, are obtained by condensing a compound of general formula—



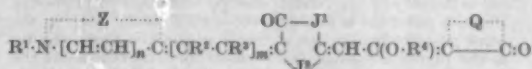
with chloral alcoholate in presence of an alkali-metal alkoxide or in presence of a basic condensing agent with a dialkoxymethyl acetate. C.O.C.

Trinuclear meroCyanine Dyes

Kodak

BP 756,208

Dyes of formula—



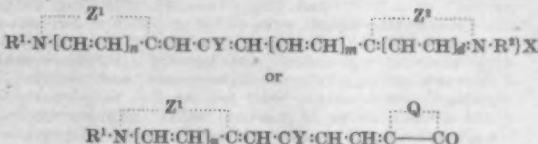
(R^1 and R^2 = Alk; R^3 = H or Alk; R^4 = H, Alk, Ar, or alkoxy; J^1 and J^2 are different, being, in either order, S and NR⁴; R^5 = subst. or unsubst. Alk or Ar, or allyl; m and n = 0 or 1; Z and Q each = atoms to complete a 5- or 6-membered heterocyclic ring, e.g. 2-[2-(3-carboxymethyl-4-oxo-2-thiono-5-thiazolidylidene)-2-ethoxyethylidene]-4-[(3-ethyl-2-benzothiazolylidene)ethylidene]-3-methyl-5-thiazolidone, have photographic sensitising properties. Syntheses of 35 examples are given. C.O.C.

Trinuclear Polymethine Dyes containing a Pyrrol, Indolyl, or Pyrrocolyl Nucleus

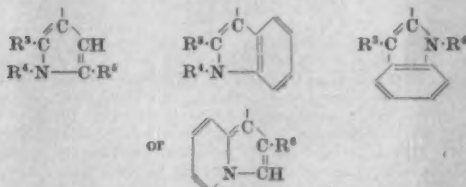
Kodak

BP 756,226

Dyes of formula—



(R¹ and R² = Alk; Z¹, Z², and Q each = atoms to complete 5- or 6-membered heterocyclic rings; X = acid radical; m, n, and d each = 0 or 1; Y =



(R³ = H, Alk, or Ar; R⁴ = H, Alk, or Ar; R⁵ = H or Alk; R⁶ = Ar), e.g. 9-(1-ethyl-2:5-dimethyl-3-pyrryl)-3:3'-dimethylthiacarboyanine iodide, are photographic sensitizers. Syntheses of 14 such dyes are given.

C.O.C.

Carbon Blacks from Aerosols

Godfrey L. Cabot

BP 757,098

Finely Divided Silica Pigment

Columbia-Southern Chemical Corp.

BP 756,857

BP 756,875

BP 756,877

BP 756,966

Fastness of the Effects of Fluorescent Brightening Agents (VIII p. 551)

V—PAINTS; ENAMELS; INKS

Rutile-type Titanium Dioxide as a Protective Pigment

J. G. Campbell and W. Hughes

J. Oil & Col. Chem. Assoc., 39, 481-482 (July 1956)

Extensive exposure trials, coupled with determinations of loss in film weight and thickness, have shown that a rutile TiO₂ subjected to complex end-treatment (Rutiox CR (British Titan Products Co. Ltd.)) protects paint media in which it is incorporated. The weight loss data satisfy the relationship—

$$R = R_0 \exp \{-KW\}$$

(R = rate of loss of weight of pigmented film; R₀ = rate of loss of weight of unpigmented film; W = weight fraction of pigment in dry film; and K = constant for the pigment under test). If pigment and binder are lost in the proportions initially present, then—

Rate of loss of film thickness

$$= R_0 \exp \{-KW\} \left(\frac{p_2 - (p_2 - p_1)W}{p_2 p_1} \right)$$

(p₁ = density of dry binder; and p₂ = density of pigment). Up to a point (at a pigment vol. concn. of ca. 25% in the work described) pigment is lost from the film more rapidly as the pigment loading is increased; beyond this point the rate of loss does not increase with further loading.

J.W.D.

Paint Application and Drying

J. J. Stordy and W. G. J. Appleton

J. Oil & Col. Chem. Assoc., 39, 565-578

(Aug. 1956)

A description of the following processes, together with a discussion of their advantages and disadvantages—dip-painting, spray-painting (hot, airless, and electrostatic methods), flow-coating, and paint stoving (by radiant heat and by convection). There are 6 photographs of plant.

J.W.D.

Polyfunctional isocyanates and Titanium Organics as Cross-linking Agents for Cellulose Derivatives

R. P. Hirt and G. N. Bruxelles

Ind. Eng. Chem., 48, 1325 (Aug. 1956)

Cross-linking agents were added to cellulose-derivative lacquers with the intention of providing coatings resistant to solvents, e.g. esters and ketones. Polyfunctional isocyanates, e.g. toluene diisocyanate and triphenylmethane trisocyanate, were too reactive with moisture and alcohols to be of practical value. Titanium esters, e.g. tetra-n-butyl titanate, caused immediate gelation on

adding to nitro- and ethyl-cellulose solutions, but titanium acetylacetonate (I) was satisfactory. Cross-linked films of reduced solvent sensitivity were obtained from cellulose nitrate, ethylcellulose, and cellulose acetate by modification with 1-10% of I. The extent of cross-linking appeared to depend on the amount of free hydroxyl in the cellulose derivative. Precoating of aluminium and steel with titanium esters did not improve the adhesion of subsequent coatings of typical cellulose-derivative lacquers.

W.K.R.

Tannins in Aniline Printing Inks

A. Angelini

Chimica (Milan), 12, 51 (1956);

J. Amer. Leather Chem. Assoc., 51, 448

(Aug. 1956)

For the preparation of printing inks, tannins are added to the solution of a basic dye in an organic solvent in the proportion of 1 mole of dye to about 4 moles of tannin; the tannin-dye lakes have a high colouring power and brilliancy, and are very resistant to light and rubbing. Several tests to find out what type of tannin is most suitable for this purpose show that the hydrolysable pyrogallol tannins are more useful than the catechol tannins, because the flavone and anthocyanin derivatives do not give good complexes with the dyes. The examination of the tannins was made by paper chromatography using a butanol-acetic acid-water mixture as a solvent, and FeCl₃, AgNO₃, and diazotised sulphanilic acid as reagents for developing the spots.

C.J.W.H.

Penetration of Ink into Paper and Its Relevance to Print Quality

R. R. Coupe and A. H. Smith

J. Oil & Col. Chem. Assoc., 39, 579-608

(Aug. 1956)

A collimated beam of light is directed at 45° on to the reverse (unprinted) side of the paper, which is supported on a glass plate, and the back-scattered light is collected by a photoelectric cell placed normally beneath the illuminated area of the paper. An ink film of known thickness may be applied to the paper surface by a mechanical, doctor-type applicator which moves at a controlled rate. As the ink penetrates the paper, there is a fall in the amount of light reflected; though the presence of a black film on the paper surface does not affect the amount of reflected light, penetration of the ink has an immediate effect. The photocell is coupled to an amplifier and thence to an oscilloscope; a photographically recorded penetration curve can thus be obtained from the moment of application of the ink. In experiments in which the effect of pressure on penetration was studied, the ink was applied by means of a small, loaded platen. The calibration of the apparatus in terms of the relationship between % print reflectance and volume ink penetration is described. The ultimate penetration of pigment and vehicle was measured by an adaptation of the section-cutting technique of Banks, Channing, and Smith (Research, 6, 348 (1953)); this is detailed. The effects of doctor blade speed, ink pressure, ink composition, and paper have been studied and the results are reported. There are six photomicrographs of paper sections showing the extent of ink penetration. "Powdering" is shown to be due not only to excessive oil absorptivity of the paper, but also equally to inability of the pigment to penetrate the paper. Depth of pigment penetration is the most important factor in controlling print density. It is important to recognise two stages in penetration—(i) the very rapid penetration during printing, due to external pressure; and (ii) the much slower one due to capillary forces within the paper-ink system. Experimental techniques are required which will simulate more closely the conditions prevailing during printing; thick ink films (which are not used in printing) provide a reservoir from which later-stage penetration is fed, whilst with thinner films there is redistribution of ink from larger to smaller pores. This may contribute to the problem of print-through or strike-through.

J.W.D.

PATENTS

Stencil Duplicating Ink

A. B. Dick Co.

BP 756,568

The ink consists of an aqueous solution of a cellulose ether or ester or an alkali-metal salt of a carboxyalkyl-cellulose containing a dissolved or dispersed colouring

matter. There should be enough cellulose derivative to give a viscosity of 35–150 sec. measured by a Stormer viscometer using a standard cup with centre baffle at 20°C. under 200 g. load.

C.O.C.

Film-forming Composition

National Lead Co.

BP 755,150

A film-forming and drying composition consists of an organic titanium ester and a vinyl resin containing 0.01–0.25 free OH group per vinyl group dissolved in a two-type solvent containing a monohydric alcohol; e.g. 1 part by wt. of tetrabutyl titanate is added to 90 parts of a 10% soln. of vinyl chloride–vinyl acetate–vinyl alcohol (90:9:1 mol.) polymer of mol. wt. 40,000 in a mixture (equal parts by wt.) of benzyl alcohol and methyl ethyl ketone. It yields coatings which withstand organic solvents, alkalis, and water.

C.O.C.

Silicone-modified Alkyd Resin Coating Compositions

Allied Chemical & Dye Corp.

BP 756,454

Treating an alkyd resin simultaneously with the low-mol. wt. products of the hydrolysis of both an aromatic silane and an aliphatic silane yields a product which produces coatings of outstanding durability, unimpaired curability, and solubility in aliphatic hydrocarbon solvents.

C.O.C.

Vivid Light-fast Organic Pigments (IV p. 543)

VI—FIBRES; YARNS; FABRICS

Underground Decomposition of Cellulosic Textiles

W. T. King

Chem. and Ind., 766–767 (28 July 1956)

Samples of cotton cloth, when buried in sandy garden soil containing 25% of water at pH 6.6 at various temp., became sufficiently tendered to be readily torn in the following times—30°C. 12 days, 50–55°C. 15 days, and 65–75°C. 30 days. Evidence of the presence of thermophilic bacteria was obtained.

A.J.

Rot-proofed Sandbags

J. M. Ashcroft and V. J. Bagdon

Amer. Dyestuff Rep., 45, 541–542, 579 (13 Aug. 1956)

Sandbags are being increasingly used for larger and more permanent military constructions. This calls for a more durable material than rot-proofed jute, and experiments have been commenced with a view to using inherently rot-proof material, e.g. cellulose acetate.

J.W.B.

Response of Animal Fibres to Acid Attack

J. Menkart and J. Detenbeck

Nature, 178, 264 (4 Aug. 1956)

Two wools, closely similar in mean diameter but differing in crimp level, were treated with acid, and their alkali solubility was determined. Stress-strain curves were also obtained. The lower-crimp wool was found to suffer greater degradation as a result of acid treatment. The difference between the two wools in their susceptibility to chemical degradation was sufficiently large to be detected in the properties of fibres dyed with metal-complex acid dyes and in the resistance of fabric to wet-flex abrasion.

W.R.M.

Fine Thermoplastic Fibres

V. A. Wentz

Ind. Eng. Chem., 48, 1342–1346 (Aug. 1956)

Extremely fine fibres (diameter 0.1–1.0 μ) have been produced from various thermoplastic materials by extruding the hot melt through fine orifices into two converging high-velocity streams of heated (700–850°F.) air. Fibre diameter is controlled by four variables—air and nozzle temperatures, air pressure, and polymer feed rate. Mats of such fibres are excellent filters for fine aerosols and smokes. Fibres of < 1 μ diameter have been prepared from nylon, poly(ethylene terephthalate), poly(methyl methacrylate), polystyrene, and polychlorotrifluoroethylene.

W.K.R.

Leuchs' [N-Carboxyglycine] Anhydride and Analogues—Some Useful Applications in Chemistry and Medicine

E. G. Curphey

Chem. and Ind., 783–785 (4 Aug. 1956)

Leuchs' anhydride (I) and its homologues, the anhydrides of N-substituted α -aminocarboxylic acids,

were used in polymerisation processes to prepare polypeptide structures by Leuchs in 1908. Recent applications include—(1) The production of polypeptide fibres; e.g. fibres of greater textile strength than nylon are obtained by the copolymerisation of I and 11-aminoundecylic acid, whereas replacing the parent anhydride by such homologues as leucine gives copolymers of lower fusion points which are useful as plasticisers. (2) The preparation of antibiotics. (3) The use of sulphur analogues, e.g. the 2-thiothiazolidones, which resemble the above anhydrides in their decomp. to the corresponding amino acids; these cpd. react smoothly with amino esters to give glycyl peptides. 17 References.

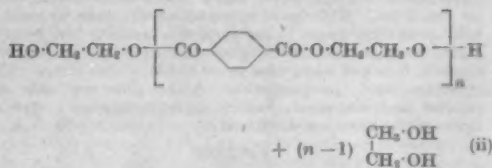
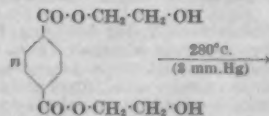
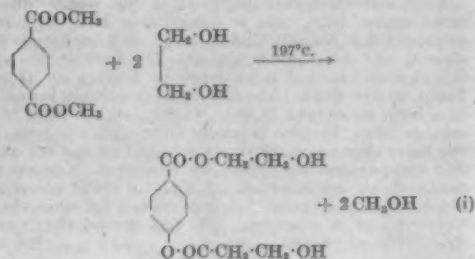
H.H.H.

Kinetics of the Polycondensation of Dimethyl Terephthalate with Ethylene Glycol—I

T. Skwarski

Zesz. Nauk. Politech. Lodz., No. 12 (*Chemia* 4), 41–65 (1956)

The polycondensation of dimethyl terephthalate with ethylene glycol takes place in two stages—



Both stages are catalysed by alkaline-earth metals or their alkoxides. The purified reagents were taken in stoichiometric proportions together with 1% of $(\text{CH}_3\text{O})_2\text{Mg}$ (on wt. of ester) as catalyst, and heated in an atmosphere of nitrogen. The by-products of the two stages of the reaction were collected separately: CH_3OH on activated charcoal at -10°C. , glycol in a U-tube filled with glass at -25°C. The first stage of the reaction was completed in 2–2.5 hr. The method for the determination of the order of reaction for the second stage of the polymerisation is described. All the calculations were based upon the wt. of ethylene glycol liberated. The values were substituted in the equation—

$$2 k t N_0^2 = \frac{1}{(1-p)^2} - 1$$

($p = (N_0 - N)/N_0$ = "degree of progress" of the reaction; N_0 = No. of functional groups initially; N = No. of functional groups at time t ; k = rate constant of the reaction). On finding that the graph of $1/(1-p)^2$ against t was a straight line, it was concluded that the reaction is of the third order. The value of k was found to be constant for a reaction of the third order. It was increased by rise of temp. within the range 240–280°C. Above 280°C. thermal decomposition takes place. The reaction proceeds much faster under reduced pressure, giving a product with a much higher D.P. All the results were checked by determination of the mol. wt. of the final products and measurement of the viscosity of the polymer dissolved in phenol

at 50°C. The work is illustrated by very informative tables, graphs, and a diagram of the apparatus used.

T.Z.W.

Physical Properties of Polyacrylonitrile Filaments

A. W. Kumm

Faserforsch. und Textiltech., 7, 365-372 (Aug. 1956)

The intermolecular bonding and orientation in polyacrylonitrile filaments are discussed, and shrinkage is considered in terms of orientation, temperature, and dimethylformamide content. The second-order transition temperature is found to be between 60° and 70°C. This, and the relatively low contribution of the elastic elongation to the total elongation, lead to flabbiness after use at 80°C.

W.R.M.

Textile Fibres from Polyvinyl Alcohol

I. Sakurada

Textil-Rund., 11, 451-458 (Aug. 1956)

Polyvinyl alcohol is dissolved in hot water, filtered, wet-spun at 70°C. into a coagulating bath of simple sulphates, e.g. Na_2SO_4 , ZnSO_4 , at 35-40°C. under 10-80% stretch, and dried under tension. The fibre at this stage is water-soluble, and is hardened by heating for 15 sec.-5 min. at 210-230°C. The fibre is subsequently formalised in a bath containing H_2SO_4 , Na_2SO_4 , and H-CHO for 40 min. at 75°C. Vinyon is produced by such a process, and the water absorption, the resistance to heat and hot water, and the physical properties are described. Vinyon C is obtained by acetalisation of Vinyon with chloroacetaldehyde, and Vinyon AN is produced by amination of Vinyon C with ammonia. The last named fibre can be dyed with acid dyes.

S.R.C.

Preparation and Properties of Glass "Semi-silk"

B. Sturm

Faserforsch. und Textiltechnik, 7, 345-357 (Aug. 1956)

Continuous glass filaments from a series of glass rods heated by gas burners are wound on a large, rapidly rotating drum to form a uniform web. This process is then interrupted, the drum is rotated in the opposite sense, and a continuous band is removed from it by means of a small roller (0.3 mm. wide) which traverses a helical path on the drum. This band is centrifugally spun to yield a new glass-fibre yarn, "glass semi-silk", which can be produced more cheaply than "glass silk", though, as its name implies, it is not so regular or lustrous as the latter. The structure and the properties of the glass semi-silk are studied and discussed. Textile manufacture and applications of the yarn are discussed.

W.R.M.

PATENTS

Jute Yarns

Ludlow Manufacturing & Sales Co.

BP 755,017

The jute fibres, before carding, are treated with water to swell them and with 4-8% (on their wt.) of a light petroleum oil, e.g. cordage oil, in which is dissolved a drying material, e.g. a drying oil. The resultant yarns are firm, full, and round with a crisp handle. Although they contain a substantial amount of oil, it will not, after drying, migrate into other yarns with which they may come into contact; e.g. they can be used as backing yarn for carpets without the oil migrating into the wool pile.

C.O.C.

Polyacrylonitrile Filaments

Vereinigte Glanzstoff-Fabriken

BP 754,775

Addition of polyvinyl acetate and polyvinyl ethyl ether to a polyacrylonitrile spinning solution results in filaments which do not readily split into fibrils.

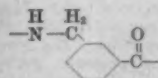
C.O.C.

Heat-resistant, Fibre-forming Synthetic Polyamides

California Research Corp.

BP 754,490

The superpolyamides of *m*-aminomethylbenzoic acid containing recurring units of formula—



are unaffected by temp. up to 320°C., have m.p. 340-370°C., and are fibre-forming.

C.O.C.

Antistatic Fabrics

Dunlop Rubber Co.

BP 756,159

Fabric composed of fibres some containing electrically conductive carbon black, the others containing no carbon black, and the proportions of the two fibres being such that the electrical resistance of the fabric is < 100,000 megohms per "unit square" at all humidities, generate no static charges by movement at any atmospheric humidity.

C.O.C.

Partial Acetylation and Cyanoethylation of Cotton

(VIII below)

Arnel Cellulose Triacetate—Properties, Applications, and Dyeing Characteristics (VIII p. 552)

Partially Acetylated Cotton Fibre and Treatments to facilitate Processing into Yarn (X p. 553)

Stabilising Polyamides (XIII p. 555).

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

PATENTS

Bleaching of Cellulose Pulps

Tennants Consolidated

BP 751,854

Sulphate pulp is bleached with Cl_2 in the presence of ClO_2 or a water-soluble chlorite in molar ratios of 2.5-3.5 : 1 or 3.5-4.5 : 1 respectively, at pH 2.5-2.7.

K.W.

Dry Cleaning

Davies-Young Soap Co.

BP 754,441

Addition to the solvent of a detergent and an antistatic agent, e.g. diethyl hydrogen phosphate, dissipates any electrostatic charges formed and renders the treated goods conductive and resistant to accumulation of electrostatic charges during drying and finishing.

C.O.C.

Dry Cleaning

FH

BP 757,075

Better cleansing of textile and leather garments is obtained by adding to the solvent a quaternary ammonium compound of high mol.wt. containing a glycol or polyglycol radical; e.g. 0.04% of hydroxyethylated octadecylamine (28 mol. of ethylene glycol) which has been methylated with dimethyl sulphate is added to carbon tetrachloride to improve its cleaning powers.

C.O.C.

Fastness of the Effects of Fluorescent Brightening Agents

(VIII p. 551)

Photochemical Oxidation of Wool in the presence of Fluorescent Compounds (VIII p. 551)

Arnel Cellulose Triacetate—Properties, Applications, and Dyeing Characteristics (VIII p. 552)

VIII—DYEING

Craft of Dyeing in Poland in the Fifteenth and Sixteenth Centuries

S. Bartkowiec

Zem. Nauk. Politech. Łódz., No. 12 (*Chemia* 4), 3-33 (1956)

The article is based on 43 works of reference, 12 of which are German. It describes the development, organisation, and working system of Polish Guilds connected with the textile industry with particular emphasis on dyeing. A number of colouring matters of the period are enumerated, and their methods of preparation and application are described.

T.Z.W.

Partial Acetylation and Cyanoethylation of Cotton

H. G. Fröhlich

Z. ges. Textilind., 58, 509-515 (5 July 1956)

The preparation and the chief properties of cyanoethylated cotton are described with detailed reference to the behaviour of the latter towards vat, direct, azoic, sulphur, disperse, acid, and basic dyes and also the influence on the nitrogen content of dyeing and other processes.

B.K.

Behaviour of Vat Dyes towards Resin Finishing

Anon.

Z. ges. Textilind., 58, 520-523 (5 July 1956)

A classification of the behaviour of Ciba and Cibacolor (Ciba) vat dyes towards treatment with Lyofix CH,

Lyofix DM, Phobotex FT-aluminium sulphate, and Pyrovatex resin finishing treatments. B.K.

Fastness to Crease-resisting of Anthrasol Dyes

G. Strohbach

Z. ges. Textilind., 58, 506-507 (5 July 1956)

A classification of the effect of crease-resist finishing on the Anthrasol (FH) vat dyes with regard to shade and fastness to light. B.K.

pH Values and their Effect on Wool in Dyeing with Irgalan Dyes in the presence of Ammonium Salts

H. R. Hirsbrunner

Textil-Rund., 11, 459-462 (Aug. 1956)

Solutions of ammonium salts show pH changes between room temperature and the boil. The trends are illustrated, based on the variation in the neutral point of pure water (pH 7.12 at 18°C. to pH 6.1 at 100°C.). The effects on disulphide sulphur and wet abrasion resistance of a number of ammonium salts are shown. An alkaline pH during raising to the boil is harmful, and for this "hot dyeing" process is advantageous. Ammonium sulphate is best, since the time of exposure to an alkaline pH is least. An excess of this salt is recommended, particularly for hard water, with corrections for the alkalinity of the fibre itself. S.R.C.

Application of Isolan Dyes (Metal-complex Dyes containing no Sulpho Groups) in Wool Dyeing

K. Wojtaschek

Melliand Textilber., 37, 81-83 (Jan. 1956)

The addition of 2% Avolan 1W to the dyebath enables level, well penetrated wool dyeings to be obtained with neutral-dyeing Isolan dyes. These dyes have no free sulpho groups and possess outstanding light fastness. Optimum dyeing pH is 4-5, which leaves the handle of the wool unimpaired. H.K.

Low-temperature Dyeing of Wool

G. H. Lister

Textil-Rund., 11, 463-466 (Aug. 1956)

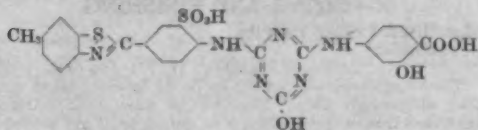
Lanasyn (S) dyes, as well as certain triphenylmethane acid dyes, can be applied substantially below the boil to yield normal depths of colour by the use of Dilatin DB (S), an emulsion of *o*-dichlorobenzene. For example, slubbing is entered, and the bath adjusted to pH 5 with acetic acid and raised to 70°C. Dilatin DB (2 g./litre) is added and circulated for 5 min.; then Sandozol N (S) (2 g./litre) is added and circulated for a further 5 min. Subsequently, the dissolved dye is added, and dyeing continued for 20 min. at 70°C. S.R.C.

Fastness of the Effects of Fluorescent Brightening Agents

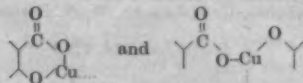
S. Bartkowiec

Zesz. Nauk. Politech. Łódz., No. 12 (*Chemia* 4), 83-97 (1956)

The theoretical possibility of obtaining fluorescent brightening agents fast to light is discussed. The agent of the following constitution was prepared—



The synthesis takes place in two stages—(i) condensation of cyanuric chloride with dehydrothio-*p*-toluidine-*o*-sulfonic acid at 0-5°C. and pH 1-5; (ii) condensation of *p*-aminosalicylic acid with the compound formed in the first stage at 20-25°C. and pH 5. "Thiazole 5" so prepared was isolated as its sodium salt. It exhibits a violet-blue fluorescence which vanishes in acid solution and is restored by strong alkali. "Optically bleached" cotton fabrics exhibited fastness to light of 1 and to washing of 3. Aftertreatment of "Thiazole 5" with Cu acetate increases its fastness to light by 0.5 and to washing by 1 grade. This is due to the formation of the following complexes—



The improvement in light fastness was associated with decrease in the brightening effect. "Thiazole 5" treated with Cu tartrate produced inferior effects. T.Z.W.

Photochemical Oxidation of Wool in the presence of Fluorescent Compounds

D. R. Graham and K. W. Statham

J.S.D.C., 72, 434-438 (Sept. 1956)

A study has been made of the action of sunlight on wool treated with fluorescent brightening agents. The discoloration which occurs is due to oxidation of the wool and appears to be independent of the fluorescent agent used. There is considerable evidence to show that oxidation of the amino acid tryptophan is the main colour-producing reaction. Authors

Influence of Different Mordants on the Colorability and Colour Intensity of Rabbit Hair

F. Stather, S. Walther, and U. Stather

Ges. Abhandl. Dest. Lederinst. Freiberg/Sa., 12, 82-86 (1955); *J. Amer. Leather Chem. Assoc.*, 51, 396 (July 1956)

Rabbit hair was dyed with Urol dyes after mordanting with one of the following mordants—(a) none, (b) $K_2Cr_2O_7$ + acetic acid, (c) as (b) + sodium acetate, (d) $K_2Cr_2O_7$ adjusted to pH 7 (monochromate), and (e) $FeSO_4$ + acetic acid. Examination of cross-sections of the dyed skins showed—(a) no dye take-up without mordant, (b) dyeing of the medulla only with iron mordant, and (c) dyeing of the cortex with Cr mordants. The optimum pH value for intensity of coloration and complete penetration was about 4. To get good penetration through the cortex the H_2O_2 used to develop the dye should be added to the dyebath after 45 min. rather than immediately. C.J.W.H.

Destructive Action of Domestic Gas-fired Driers on Certain Dyes applied to Cotton

AATCC Midwest Section

Amer. Dyestuff Rep., 45, P 471 (16 July 1956)

Following complaints that cotton materials dyed with dyes considered fast to washing faded in domestic gas-fired driers, a laboratory procedure has been devised which predicts the sensitivity of such dyes. The trouble is not confined to any group of dyes, and is due to the action of oxides of nitrogen on wet material. The test fabric is dipped into a solution containing 0.4 g. of sodium nitrite and 0.4 c.c. of glacial acetic acid per litre, squeezed by hand, and pressed dry between filter papers. This is repeated ten times, and the specimens are thoroughly rinsed in running cold water to remove traces of sodium salts, and pressed dry. The only dyes affected to a significant extent were blues of the chlorinated indanthrone type (C.I. 1112 and 1113) and mixture dyeings containing them. Restoration of the colour may be effected by treating with a mild hydrosulphite bath. J.W.B.

Current Developments in the Application of Dyes

L. S. Thompson

Amer. Dyestuff Rep., 45, P 544-P 547 (13 Aug. 1956)

A brief survey, mainly of the dyeing of newer fibres. J.W.B.

Recent Developments in the Dyeing of Synthetic Fibres

A. Würz

Melliand Textilber., 37, 83-90 (Jan. 1956)

A survey of modern techniques in the dyeing of polyamide, polyacrylonitrile, and polyester fibres is given with 33 references to the literature. Some information on polyvinyl chloride and polyvinyl alcohol is also given together with information on the dyeing of acrylonitrile-vinyl chloride copolymers. H.K.

Dyeing of Synthetic Fibres with Disperse Dyes

F. Weber

Z. ges. Textilind., 58, 516-518 (5 July 1956)

A review of various methods of applying dyes to synthetic fibres together with a classification of Celliton (BASF), Cibacet (Ciba), Latyl (DuP), Acetoquinone (Fran), Setacyl (Gy), Solacet, Dispersol and Duranol (ICI), and Artisil (S) dyes with respect to sublimation, high-temperature dyeing, and suitability for dyeing polyvinyl chloride, polyamide, acrylic, polyester, and cellulose triacetate fibres. B.K.

Arnel Cellulose Triacetate—Properties, Applications, and Dyeing Characteristics

A. F. Tesi

Amer. Dyestuff Rep., **45**, P 512-P 517
(30 July 1956)

A survey is briefly presented of the properties of Arnel (Celanese Corp. of America) triacetate fibre, covering strength, thermal behaviour, dyeing with and without "accelerants", laundering shrinkage and strength retention, and rate of saponification under winch scouring conditions. The importance of correct heat-treatment is emphasised. Some information is given on the dyeing of blends containing Arnel.

J.W.B.

Dyeing and Related Processing of Nylenka (6-Nylon)

R. M. Stribling

Amer. Dyestuff Rep., **45**, P 452-P 455
(2 July 1956)

Manufacture, characteristics, and processing of Nylenka (Enka) are discussed, the processing covering blends with viscose rayon staple (including carpets), full-fashioned hosiery, and socks containing stretch yarns.

J.W.B.

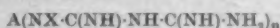
PATENTS

Imparting Affinity for Acid and Mordant Dyes to Cellulose and Improving the Affinity for Dyes of Polyamide Fibres

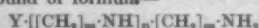
ICI

BP 755,478

Cellulose treated, e.g. at 50–100°C., in an aqueous solution of the water-soluble product obtained by heating together in absence of water a salt of a compound of formula—



(A = H, Alk, alkylene, aralkyl, Ar, or arylene which may contain one or more neutral or basic substituents; X = H or Alk; n = 1 or 2), e.g. phenyl diguanide carbonate, with a compound of formula—



(Y = NH₂, OH, or Hal; m = 2–6; p = a whole number), e.g. tetraethylenepentamine, can be dyed with acid or mordant dyes. Polyamide fibres so treated take up several times more than untreated fibres of dyes solubilised by anionic groups.

BP 755,519

Products obtained by heating together in absence of water a compound of formula A(NX·C(NH)·NH·CN)_n with one of formula Y·[(CH₂)_m·NH]_p·[CH₂]_m·NH₂ are similarly used.

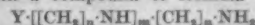
C.O.C.

Improving the Dyeability of Polyamide Fibres

ICI

BP 755,520

Polyamide fibres treated at 50–100°C. with the products obtained by reaction of a diacydiamide, cyanamide, or guanidine with a compound of formula—



(Y = NH₂, OH, or Hal; n = 2–6; m = whole number), e.g. diethylenetriamine, absorb several times as much of a dye solubilised by an anionic group as do untreated fibres.

C.O.C.

Response of Animal Fibres to Acid Attack (VI p. 549)
Anionic Dyes in Leather Colouring (XII p. 555)

IX—PRINTING

Historical Notes on the Wet-processing Industry XII—Calicoes and Indiennes—Eighteenth-century Printing

S. M. Edelstein

Amer. Dyestuff Rep., **45**, 495–500 (16 July 1956)

Practical Interpretations of the Results of Laboratory Investigations in the Field of Vat Printing

M. R. Fox

Amer. Dyestuff Rep., **45**, P 447-P 451
(2 July 1956)

Factors influencing the reduction and fixation of printed vat dyes are outlined, and a number of graphs are shown to illustrate the relation between sulfoxylate concentration, electronegative potential, time of storage, drying temperature, duration of steaming, and percentage fixation.

J.W.B.

Special Problems in Vat Printing Practice

A. Garscha

Melliand Textilber., **37**, 91–93 (Jan. 1956)

The preparation of vat printing pastes can be made a relatively simple routine, particularly by using the Votator (Girdler Corp.), the boiler apparatus which will prepare 4–500 kg. of print thickening per hour. The suitability of some vat dyes is referred to.

H.K.

PATENTS

Fabric Printing

STAR Stampa Tessuti Artistici

BP 754,233

A multicoloured pattern is printed on a non-stretchable carrier, e.g. of paper, which has been coated with an aqueous solution of sodium palmitate, castor-oil super-sulphonate, and zinc stearate. When the pattern is intaglio-printed on the carrier the dyes used must be organic and are applied from solution in a mixture of toluene, Albertol (m.p. 105°C.: see *Synthetic Resins and their Plastics*, by Ellis (1923), pp. 147–148), paraffin, and sodium polysulphonicoleate. The pattern is then transferred to the fabric by means of heat, pressure, and solvents.

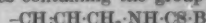
C.O.C.

Diazotype Materials

General Aniline

BP 756,308

If the sensitive layer contains thiourea or thiocarboxamide derivatives containing the group—



(the ethylenic group forms part of either an open hydrocarbon chain or an aromatic ring of the benzene or

naphthalene series; B = R-NH- or -N-Z (R = Alk, hydroxyalkyl, or alkoxyalkyl; Z = atoms to complete a saturated heterocyclic ring), e.g. 1-allyl-3-β-hydroxyethyl-2-thiourea, then the rate of azo dye development with low ammonia concentration is greatly accelerated.

C.O.C.

Spot-carbonising of Paper by Typographic Printing

Arthur J. Gavrin Press

BP 754,547

A normally solid carbon ink is melted, and while molten is fed to a heat-insulating resilient transfer roller, which passes it on to a heat-insulating resilient typographic plate for printing.

C.O.C.

Xerographic Plate

Haloid Co.

BP 755,683

Electrostatic Printing

Horizons

BP 755,486

Colour-corrected Multicolour Photographs

Agfa

BP 755,458

Colour Development in Colour Photography

Kodak

BP 755,655

Colour Couplers (IV p. 543)

Penetration of Ink into Paper and its Relevance to Print Quality (V p. 548)

X—SIZING AND FINISHING

Metallised Lining Fabrics

A. Harenslak

Melliand Textilber., **37**, 101–103 (Jan. 1956)

Tests have been carried out on lining fabrics coated with thin aluminium films. The results show that infrared reflection from a dyed fabric is as strong as, if not stronger than, from a metallised fabric, that metallisation has no influence on the thermal transmission, and that air permeability is considerably reduced. Coating a lining fabric with metallic powder does not improve the serviceability of the lining.

H.K.

Cotton Finishing

H. C. Borghetty

Amer. Dyestuff Rep., **45**, P 518-P 523
(30 July 1956)

A brief survey of cotton finishing, with emphasis on modern chemical techniques, particularly the application of thermoplastic and thermosetting resins.

J.W.B.

Cyanoethylation of Cotton Fabric

L. H. Greathouse, H. J. Janssen, W. N. Berard, and C. H. Haydel

Ind. Eng. Chem., **46**, 1263–1267 (Aug. 1956)

A pilot-plant study of the cyanoethylation of cotton fabric is reported. Fifty-yard lengths of a light-weight

print cloth were uniformly cyanoethylated within limits of 0.40–0.42 cyanoethyl group per anhydroglucose unit, by careful control of reaction conditions, particularly temperature. Under the best conditions the uniformity extended to individual fibres, except for slight deviations at the extreme ends of the roll. It is shown that avoidance of local overheating in the exothermic reaction is crucial in producing uniform treatment. W.K.R.

Chemical and Physical Properties of Viscose Rayon Continuous Filament treated with Solutions of Bishydroxymethylurea (Dimethylolurea)

A. Nowakowski

Zesz. Nauk. Politech. Łódz., No. 12 (*Chemia* 4), 67–81 (1956)

The swelling of continuous-filament viscose rayon spun with the elimination of stretch can be decreased by treating it with dimethylolurea resin, prepared from 1 part of urea and 2 parts of formaldehyde, with NaOH as catalyst. It was found that the resin solution diffuses inside the filament, forming a carbamide compound in the final stages of the condensation. Better results were obtained by using wetted-out filament, higher concentration of resin solution in the impregnating bath at pH 4, and a higher temperature of the final stage of the condensation. The effect produced is resistant to ten severe washings. T.Z.W.

Finishing highly Encrusted Natural Plaiting Fibres—Straw, Bamboo, Pandanus, Panama, and Palm Fibres

A. Gebhardt

Melliand Textilber., 37, 103–104 (Jan. 1956)

Use of Acrylate Resins in Crease-resistant Finishes

R. Steele and C. L. Browne

Amer. Dyestuff Rep., 45, 525–528 (30 July 1956)

About 1% of soft acrylate resin, applied as a dispersion, produces an increase of 10–20% in the crease recovery of cotton fabrics, the effect being additive to that produced by thermosetting resins. It can be observed on the fabric in wet as well as dry crease-recovery tests, which suggests that the acrylate would also contribute to the wash-wear characteristics of treated fabric. The reagent may be particularly useful where only moderate crease resistance is required or where strength loss due to treatment with thermosetting resins is excessive. J.W.B.

Chemical Considerations of Woollen and Worsted Finishing

H. C. Haller

Amer. Dyestuff Rep., 45, P 484–P 490 (16 July 1956)

An outline is given of the chemical side of wool finishing, covering shrink resistance, mothproofing, showerproofing and crease-resistance, and softening. J.W.B.

Partially Acetylated Cotton Fibre and Treatments to facilitate Processing into Yarn

W. G. Sloan, E. M. Buras, C. F. Goldthwait, and A. L. Murphy

Amer. Dyestuff Rep., 45, 429–433 (2 July 1956)

Physical and chemical changes brought about by acetylating cotton to 18–21% acetyl content are discussed. Trouble due to static in processing can be reduced by various additives, most of which, however, lead to lower heat-resistance. Two antistatic agents are found (but not named) which do not have this fault. Blending with 20% of untreated cotton also results in satisfactory control. J.W.B.

Finishing of Fabrics containing Terylene Polyester Staple Fibre

N. M. Mims

J.S.D.C., 72, 426–430 (Sept. 1956)

This paper describes some of the differences between the properties of Terylene polyester fibre and wool, discusses how these differences manifest themselves in finishing, and outlines the response to finishing treatments of fabrics containing Terylene staple fibre, both by itself and blended with botany wool. AUTHOR

Silicones and their Application in Textile Finishing—I

W. Weltzien and G. Hauschild

Melliand Textilber., 37, 199–203 (Feb. 1956)

The development of silicones and their application to textiles either from solution or as emulsions has produced

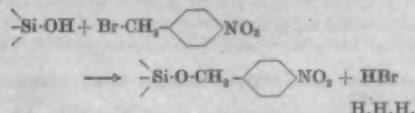
a new range of finishes, of which the most important is probably the water-repellent. The determination of silica by oxidation of the silicones with sodium peroxide in a bomb is described together with details of the construction of the bomb and ancillary apparatus. H.K.

Modification of Glass Surfaces by *p*-Nitrobenzyl Bromide

W. Haller and H. C. Duecker

Nature, 178, 376–377 (18 Aug. 1956)

Glass surfaces become permanently hydrophobic after treatment for 1 hr. at 100°C. with a dry 30% toluene soln. of *p*-nitrobenzyl bromide. The effect is found with vitreous silica, borosilicate glasses, and glasses of other compositions, and is most pronounced with surfaces which have been freshly cleaved under distilled H₂O and vacuum-dried. Similar treatment with aq. cetylpyridinium hydrochloride produced hydrophobicity which was unstable towards distilled H₂O. The stability of the former treatment suggests the presence of an organic surface derivative of the glass rather than an ion-exchange or adsorption layer as produced on glass surfaces treated with quaternary ammonium epd. Reactivity appears to depend on the –CH₂Br group, since no degree of hydrophobicity is produced by *p*-nitrotoluene itself. Glass surfaces with the largest number of OH groups per unit surface area are the most efficient, and the mechanism proposed is the formation of a deriv. of the surface OH of the glass by the reaction—



H.H.H.

PATENTS

Voluminous Yarn

Celanese Corp. of America

BP 755,580

Continuous-filament yarn is twisted while plastic, the twist removed, and the yarn relaxed to a degree less than that by which it would shorten if allowed to relax freely in absence of tension. This improves the crimp, voluminosity, and elasticity, while the breaking load and elongation are nearer to those of the untreated yarn. C.O.C.

Crimped Yarn

G. Coudéne

BP 755,597

Yarn, particularly yarn of polyamide filaments, is first highly twisted, then back-twisted to its initial degree of twist, and finally heated in absence of tension to develop crimp. C.O.C.

Yarn Sizing

Gebrüder Sucker

BP 754,798

The dry yarn is squeezed, and then immediately after squeezing and without being exposed to the air it is drawn under tension into a bath of size and squeezed again before it is withdrawn from the size. C.O.C.

Reducing the Water Absorbency of Polysaccharide Fibrous Materials

National Lead Co.

BP 754,473

The materials are treated with a solution of a Ti or Zr compound and an aldehyde or ketone. Thus cotton cloth was treated for 5 min. in an aqueous solution of TiOSO₄ (10 g. TiO₂ per litre) and glyoxal (10 g. per litre) at 25°C., squeezed until it contained its own weight of liquor, dried for 10 min. at 60°C., immersed for 30 min. in 1% aq. Na₂CO₃, rinsed, and dried. The treated cloth was dimensionally stable and had good crease-shedding properties. C.O.C.

Water-repellent Finish on Cellulosic Fabrics

Midland Silicones

BP 755,028

A cellulosic fabric is impregnated with an organosilicon compound, compressively shrunk, and then heated to <100°F. until the organosilicon compound is cured. C.O.C.

Flame-resistant Pile Fabrics

Celanese Corp. of America

BP 755,022

Fabric containing an organic derivative in the pile is impregnated with an aqueous solution of borax, either borax or sulphamic acid, and a non-ionic or anionic waxy softening agent. Thus a carpet having a cellulose acetate pile does not propagate flame if it has been sprayed with a

mixture of borax (4 parts), boric acid (4), Ahcovel R (0.2), and water (91.8) at 50°C. so that there is 6.4 oz. per sq. ft. of carpet, and then dried at 200°F. C.O.C.

Embossing Thermoplastic Fabrics

BrC BP 755,632
The fabric is wetted with a liquid which acts as a softening agent only at high temperatures. It is then sucked on to a heated perforate pattern by an elastic membrane, suction being exerted through the perforations in the pattern and the interstices of the fabric. Finally it is cooled before it is removed from the pattern. C.O.C.

Coating Fibrous Glass with Metal

Owens-Corning Fiberglass Corp. BP 755,719
Glass textiles can be continuously coated by introducing chemical reagents on to them as they are fed into the apparatus. The reagents are retained in the interstices of the material by capillary action, and the chemical reaction to deposit the metal is allowed to occur *in situ*. This yields complete coating of the fibres. C.O.C.

Bonding with isocyanate-modified Polyesters

FBy BP 756,076
Modification of BP 692,045 (cf. J.S.D.C., 69, 311 (1953)) and 700,617. If in the modified polyester the amount of diisocyanate used is in excess of that capable of reacting with the end-groups of the polyester, the bonding strength is increased and use of a cross-linking agent is unnecessary. C.O.C.

Rot-proofed Sandbags (VI p. 549)

Partial Acetylation and Cyanoethylation of Cotton (VIII p. 550)

Behaviour of Vat Dyes towards Resin Finishing (VIII p. 550)

Fastness to Crease-resisting of Anthrasol Dyes (VIII p. 551)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

New Paper Products in Competition with Textiles R. B. Hobbs

Amer. Dyestuff Rep., 45, P 479-P 481, P 483 (16 July 1956)

Behaviour of Cellulose at Different Temperatures H. Demus

Faserforsch. und Textiltech., 7, 357-365 (Aug. 1956)

The literature concerning the behaviour of cellulose at different temperatures and measures to improve its heat stability is reviewed. Measurements are made of resistance to tearing, extension, viscosity in cuprammonium, and swelling of untreated filaments and filaments washed in water and in dilute alkali, after heating at different temperatures for varying times. Chemical variations are estimated from copper numbers. Deterioration of cellulose due to heat is attributed to decreasing stability of the cellulose molecule and to external influences. The lowest ignition temperature was found to be 290°C. W.R.M.

Stabilisation of Ethylcellulose against Photo-oxidation

J. B. Batdorf and G. M. Gantz.

Ind. Eng. Chem., 48, 1325 (Aug. 1956)

Incorporation of an ultraviolet absorber (0.5% of 2,4-dihydroxybenzophenone) together with an antioxidant (0.5% of *p*-tert.-amylphenol) effectively stabilises plasticised ethylcellulose films against degradation on exposure to light and weathering. W.K.R.

PATENTS

Highly Finished Paper Products

J. C. Rice BP 751,729

Paper board is coated by conventional coating methods with a composition consisting of ~ 50% by wt. of clay and/or white pigment, and 15-25% on the wt. of clay of a thermoplastic resin (which may contain synthetic rubber) and a protein or carbohydrate adhesive. The web is subsequently dried to a moisture content of 5-15% and is then cast-moulded, i.e. a cotton roll presses the coated side against a heated polished roll, the moulding pressure being about 1-3 tons per lineal inch, to produce a smooth and high-gloss surface. K.W.

Coating Paper with Viscous Aqueous Solutions

DuP BP 755,151

The paper, while travelling at < 150 ft./min. and 1-200 times "faster than" the linear speed of extrusion of the composition, is coated with the latter at 75-150°F. and under a pressure of 20-190 mm. of mercury in absence of non-condensable gases but in the presence of added water vapour or readily condensable vapours of an inert, water-insoluble liquid of b.p. > 212°F. at atmospheric pressure. This yields extremely thin coatings of uniform thickness, free from entrapped bubbles. C.O.C.

Manifold Paper

Caribonum BP 757,136

The paper carries an alkaline substance and a colourless dye either which becomes converted into the dye when the alkaline substance is neutralised. The alkaline substance and the acid may both be carried on the paper but kept apart by a barrier layer until pressure is exerted on the paper. C.O.C.

Penetration of Ink into Paper and its Relevance to Print Quality (V p. 548)

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Atmospheric Oxidation of Alkali Sulphide Solutions and its Effect on the Unhairing and Dewooling of Skins

H. Booth

J. Soc. Leather Trades Chem., 40, 226-239 (July 1956)

Alkali sulphide soln. are oxidised by air by two different mechanisms: (1) in less alkaline soln. of NaHS or (NH₄)₂S with limited O₂ supply, polysulphide formation; (2) with good aeration or more alkaline soln., a reaction catalysed by Fe. Reaction (1) is slow, complete oxidation taking a few days or weeks. Reaction (2) can be extremely rapid in alkaline soln. with good aeration or on a surface where air has free access, complete oxidation taking < 1 day. Both reactions normally yield thiosulphate, but with reaction (2) in alkaline soln. one-third of the sulphide can be oxidised to sulphate if there is a restricted supply of O₂. An account is given of the mechanism of the catalytic oxidation, which involves the formation of Fe₂S₃, the ferric salt of a complex anion FeS₃²⁻. Some of the iron can be present as complex sulphides which are without catalytic action. The consequences of the oxidation of Na₂S in the presence of lime by the Fe present in all types of skin are discussed. J.W.D.

Reaction of Nitrous Acid with Collagen

J. M. Cassel

J. Amer. Leather Chem. Assoc., 51, 406-417 (Aug. 1956)

The reaction between HNO₂ and collagen at 16°C. has been studied by determining the amino acid composition (as revealed by two-dimensional paper chromatography) of derivatives prepared by varying the reaction time from 5 min. to 6 days. One sample, after reaction with HNO₂, was decolorised with permanganate and bisulphite. Aspartic acid, glutamic acid, glycine, proline, hydroxyproline, leucine, isoleucine, valine, alanine, phenylalanine, and histidine are not attacked by HNO₂. The ϵ -amino, guanidino, and phenolic groups of lysine, arginine, and tyrosine respectively are attacked, but not to the same extent. The lysine content decreases progressively to zero. In samples deaminated for > 12 hr. the arginine content is reduced. The tyrosine content decreases progressively with deamination, and both methionine and tyrosine are attacked by the decolorising process. J.W.D.

Investigations on Bating. I—A Historical and Critical Review of the Literature

G. H. Green

J. Soc. Leather Trades Chem., 40, 195-204 (June 1956)

The literature on the mechanism of bating is reviewed historically. Although numerous theories have been proposed, none has been universally accepted as explaining all the known effects of bating on the pelt and on the resulting leather. J.W.D.

Anionic Dyes in Leather Colouring

J. A. Camacho

Leather Mfr., 73, (4), 24-38 (1956);
J. Amer. Leather Chem. Assoc., 51, 455
 (Aug. 1956)

An outline of the anionic dyes of the azo, triarylmethane, polyheterocyclic, and anthraquinone classes, including a brief history of the various classes, methods of preparing the dyes in the group, their structural formulae, and colour. In some cases the properties of the dyes are given with respect to fastness, rubbing, penetration, and how various salts affect the colour. Also discussed are the good and bad features of the dye or group, the pH of 0.2% soln. of dyes, and how temperature affects some dyes.

C.J.W.H.

Covering Power of Pigments used in Leather Finishing

E. Belavsky and T. Termignoni

Rev. Quim. Industr., Rio de Janeiro, 24, 21 (1955);
J. Appl. Chem. Abs., 11, 824 (1955);
J. Soc. Leather Trades Chem., 40, 244 (July 1956)

For good finishes, particle size of the pigment must be less than the thickness of the film, and pigment must be air- and light-fast. Covering power depends on the comparative refractive indices of pigment and medium. Three general bases are used for leather finishing—casein, cellulose nitrate, and polyacrylic or polyvinyl resins. Tests were made using a number of combinations of medium and pigment. Titanium oxide, with its high refractive index (2.30-2.46), has high covering power in all media, especially synthetic resins, and may be cheapened by mixing with barium sulphate. A table gives pigments and media for leather finishes of various colours.

C.J.W.H.

Metallic Stains on Leather

J. J. Tancous and F. O'Flaherty

J. Amer. Leather Chem. Assoc., 51, 350-352
 (July 1956)

Metallic stains can be caused at many stages in the handling of skins and hides, and may not become apparent until some later process causes a chemical reaction with the metal. The formation, identification, prevention, and removal of such stains are briefly discussed. J.W.D.

PATENTS

Tanning Agents

CFM

BP 756,481

The products obtained by condensing hydroxymethyl compounds of diacyandiamide and/or of diacyandiamidine with polyhydric phenols in acid medium at < 50°C. when used in tanning yield leather of light colour which can be dyed and greased like chrome-tanned leather. C.O.C.

Wet Treatment of Animal Skins

Seootan

BP 755,960

The skin is placed on a porous bed and then a thin flexible, preferably elastic, membrane is laid over the entire bed, and fluid pressure is applied to the membrane to force it against the bed. The treating liquor is then introduced under pressure between the skin and the membrane, this pressure being sufficient to cause the liquor to cover the skin but insufficient to separate the membrane from the parts of the bed not covered by the skin, the pressure on the membrane being kept up during the whole of the treatment. C.O.C.

Tanning

Boehme Fettchemie

BP 755,331

Emulsions made by the process of BP 753,668 (cf. J.S.D.C., 72, 493 (Oct. 1956)), using a polyalkylene oxide wax of mol. wt. < 2000, a non-sulphonated oil, and an inorganic tanning agent, are particularly suitable for a combined brush-tanning and fatting of hides. They are very stable and may be added to a chrome tanning liquor. Their use gives full-handling leathers without need for separate fat-liquoring. C.O.C.

Leuchs' [N-Carboxyglycine] Anhydride and Analogues—
 Some Useful Applications in Chemistry and Medicine
 (VI p. 549)

Influence of Different Mordants on the Colorability and
 Colour Intensity of Rabbit Hair (VIII p. 551)

Selective Stain for Elastic Tissue (XIV p. 556)

XIII—RUBBER; RESINS; PLASTICS**Effect of Temperature on the Cross-linking of Polyethylene by High-energy Radiation**

R. M. Black

Nature, 178, 305-306 (11 Aug. 1956)

Studies of the cross-linking induced in polyethylene by high-energy radiation over a wide temperature range show that less energy is required to produce a cross-link as the temperature of irradiation is increased. The mechanism of cross-linking is briefly discussed. W.R.M.

PATENTS

Improving the Adhesive Properties of Polyethylene Surfaces

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The surface of polyethylene is given improved adhesive properties, e.g. for printing inks, if, as the polyethylene is extruded at < 150°C., it is passed through an aqueous solution of a halogen, hydrogen halide, H₂O₂, HNO₃, alkaline hypochlorite, conc. HNO₃, or a mixture of conc. HNO₃ and conc. H₂SO₄, at 25-95°C., preferably in presence of ultraviolet radiation. C.O.C.

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Decorated Thermosetting Synthetic-resin Articles

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Well felted paper impregnated with a bonding medium has a pattern printed on it with a colour which is unaffected by heat. It is then impregnated with a thermosetting synthetic resin with simultaneous removal of air contained in the paper. The resin is partly condensed to render the paper hard and brittle, moisture is applied to render the paper pliable; then the paper is placed with its printed side in contact with a supply of incompletely condensed thermosetting synthetic resin to form the object, and is then heated and pressed to complete condensation and unite the two components. C.O.C.

Marbled Thermoplastic or Rubber-composition Sheets

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BP 756,383

Stabilising Polyamides

Perfogit

BP 756,384

Addition of butyrolactone in the preparation of polyamides results in improved products and permits better control of the mol. wt. of the polyamide. C.O.C.

XIV—ANALYSIS; TESTING; APPARATUS**Analytical Reviews**

Anal. Chem., 28, 559-782 (April 1956, Part II)

Annual review of progress in analytical chemistry, under 29 broad headings, with 5659 references to the literature. For the first time the review is presented in a separate volume. J.W.D.

Rôle of Molecular Complexes in Chromatographic Adsorption

S. Basu

Chem. and Ind., 764-765 (28 July 1956)

Evidence is presented that molecular complexes are formed in the adsorption of molecules on a solid surface. Complex formation is facilitated by high electron affinity of one constituent and low ionisation potential of the other. In comparing relative ease of adsorption of a number of compounds the energy of the topmost filled orbital of the conjugated organic molecule, calculated by the L.C.A.O. method, may be used in place of ionisation potential. Results for some polyacenes and diphenylpolyenes are given in support. A.J.

Thermochromism, a Possible Source of Error in Colorimetric Analysis. Experiments with 3:5-Dinitrosalicylic Acid

R. T. Bottle and G. A. Gilbert

Chem. and Ind., 575-576 (30 June 1956)

3:5-Dinitrosalicylic acid exhibits thermochromism in

alkaline soln. This may lead to large errors when the reagent is used to determine reducing sugars or reducing end-groups in polysaccharides. Thermochromism is thought to be due to interaction between the ionised carbonyl and the *ortho*-nitro group, since it is much diminished for the free acid in ethanol or the reduced compound. A.J.

Indicator for Titration of Calcium in Presence of Magnesium using Disodium Dihydrogen Ethylenediaminetetra-acetate

H. Diehl and J. L. Ellingboe

Anal. Chem., **28**, 882-884 (May 1956)

A new indicator, given the trivial name *calcein*, has been prepared for the titration of Ca in the presence of Mg with disodium dihydrogen ethylenediaminetetra-acetate; it is the condensation product of iminodiacetic acid and fluorescein. It is necessary only to dissolve the sample and to adjust the pH to 12; no other preliminary treatment is required. Large excesses of Na and Mg cause the results for Ca to be slightly low. Cyanide is added to complex Cu and Fe if they are present. Calcein gives a sharper end-point than does Eriochrome Black T and tolerates larger amounts of Mg and Na without impairing the end-point. J.W.D.

Determination of Triphosphate in Commercial Triphosphate and Detergents built with Triphosphate

H. J. Weiser

Anal. Chem., **28**, 477-481 (April 1956)

The triphosphate ion $P_3O_{10}^{5-}$ is pptd. from acid soln. by the trivalent triethylenediaminecobalt^{III} ion. The method has a standard deviation of 0.8% absolute, and a reproducibility of 0.5% absolute; this compares favourably with the isotope dilution method, for which these data are 1.5% and +2% absolute respectively. Pyrophosphate tends to coprecipitate, but this is compensated for by the use of a calibration curve and carefully controlled pptn. conditions. Appreciable amounts of phosphate glass interfere, but the method gives warning of such interference. J.W.D.

Analysis of Phosphorus Compounds—Rapid Hydrolysis of Condensed Phosphates in Volumetric Analysis

E. J. Griffith

Anal. Chem., **28**, 525-526 (April 1956)

The time required for the hydrolysis of condensed phosphates to the ortho form has been reduced from a minimum of 8 hr. to < 1 hr. by boiling the sample to dryness from strongly acidic soln. The addition of alkali-metal chlorides prevents the recondensation of the orthophosphate during the heating to dryness. J.W.D.

Use of Ionic Dyes in the Analysis of Ionic Surfactants and Other Ionic Organic Compounds

P. Mukerjee

Anal. Chem., **28**, 870-873 (May 1956)

A partition technique for the analysis of all classes of ionic surfactants and similar organic cpd. is presented and the theory is discussed. The sensitivity of the method is very high, and quant. estimations of concn. of the order of 0.1-1 p.p.m. and amounts of the order of 0.001-0.01 mg. are possible. Hydrolysable and non-hydrolysable surfactants in mixtures may be determined and numerous examples are given. J.W.D.

New Method of Testing the Emulsion Stability of Fats with a Mineral-oil Basis

R. Schiffrer

Faserforsch. und Textiltech., **7**, 374-376 (Aug. 1956)

100 ml. of mineral oil fat and 400 ml. of water are mixed, and samples of definite volume withdrawn into a graduated centrifuge tube immediately and then at noted times after mixing. Conc. $NaNO_3$ soln. is added to the samples, which are heated on a water-bath for 20-30 min. and then centrifuged. Stability is estimated from the volume of the oily layer at different times after mixing. The method is applied also to mineral-oil emulsions containing emulsifying agents. W.R.M.

Partition Chromatography of Aliphatic Acids—Quantitative Resolution of Normal-chain Even Acids from C_{12} to C_{24}

F. A. Vandenheuvel and D. R. Vatcher

Anal. Chem., **28**, 838-845 (May 1956)

Qualitative and Semi-quantitative Tests for Amine Hydrochlorides on Paper Chromatograms

S. D. Nogaro

Anal. Chem., **28**, 903 (May 1956)

The chloride ion is detected by its reaction with Ag^+ . Qualitatively, the amine hydrochloride spots are detected by the reduction of $AgCl$ to metallic Ag. In the quant. test the Cl^- content of the spots from a separate chromatogram may be titrated by the sensitive potentiometric procedure of Kolthoff and Kuroda (*ibid.*, **23**, 1304 (1951)). Spots which contain as little as 3 $\mu g.$ of Cl^- may be qual. located. The quant. test shows ca. 88% recovery, with good precision. The tests do not appear to depend in any way upon amine structure, but require simply that the amine is in the hydrochloride form. J.W.D.

Pyrocatechol Violet as a Detection Reagent for Cations

K. Macek and L. Morávek

Nature, **178**, 102-103 (14 July 1956)

Pyrocatechol Violet forms coloured complexes with many cations of valency 3-6: Al^{3+} , Fe^{3+} , Sb^{3+} , Bi^{3+} , Th^{4+} , Zr^{4+} , V^{5+} , Cr^{6+} , Mo^{6+} , W^{6+} give the best reaction and may be detected on chromatograms down to 0.1 $\mu g.$ The reagent is applied to chromatograms or electropherograms as a 0.05% soln. in ethanol; acid conditions are optimal, though alkaline media are sometimes of value, since they enable some bivalent cations (Co^{2+} , Ni^{2+}) to be detected which react only slightly in acid media. J.W.D.

Determination of Copper in Textiles

A. Mutschin

Melliand Textilber., **37**, 104-105 (Jan. 1956)

The method depends upon digestion of the sample by a Kjeldahl technique using conc. H_2SO_4 and $NaNO_3$ to remove organic matter, followed by electrolytic determination of the copper. Large-scale analysis of copper-containing textiles can be simplified by direct Schwarzenbach titration after Kjeldahl digestion. H.K.

Photometer for Measuring Cloth Irregularity

M. Magalhães, D. A. Harrison, and W. J. Onions

J. Textile Inst., **47**, p. 481-p. 485 (July 1956)

An instrument is described for measuring the variation in reflectance of a cloth, and it is found that the coefficients of variation of reflectance are closely correlated with the appraisal of cloth by the human eye. Subjective preferences may, however, be influenced by periodicity as well as amplitude of yarn variation, and the simple coefficient of variation of reflectance will not account for this. Surface hairs and aperture size influence the coefficient of variation strongly. For fabrics knitted from a series of 18 worsted yarns differing in mean twist and irregularity, a highly significant correlation is found between the variation of fabric reflectance and the yarn irregularities. J.W.B.

Amperometric Titration of Viscose

M. Wronski

Faserforsch. und Textiltech., **7**, 372-374 (Aug. 1956)

Apparatus and methods for amperometric estimation of sulphide, thiocarbonate, and xanthate in viscose are described. W.R.M.

Selective Stain for Elastic Tissue

H. M. Fuller and R. D. Lillie

Stain Technol., **31**, 27-29 (1956);

J. Amer. Leather Chem. Assoc., **51**, 401 (July 1956)

A selective stain for elastic tissue (designated oreinol-new fuchsin) is described. Two grams of new fuchsin (C.I. 678) and 4 g. of oreinol (highest purity) are added to 200 ml. of distilled water, and the solution is boiled for 5 min. Then 25 ml. of $FeCl_3$ soln. (U.S.P. IX) is added, and the solution is boiled 5 min. longer. The precipitate is collected and dissolved in 100 ml. of 95% ethanol. This is the staining solution. Sections are deparaffinised and brought to absolute ethanol, stained for 15 min. at 37°C. with oreinol-new fuchsin, differentiated for 15 min. in 70% ethanol, cleared, and covered as usual. C.J.W.H.

Tannins in Aniline Printing Inks (V, p. 548)

Destructive Action of Domestic Gas-fired Driers on Certain Dyes applied to Cotton (VIII p. 551)

Silicones and their Application in Textile Finishing—I (X p. 553)

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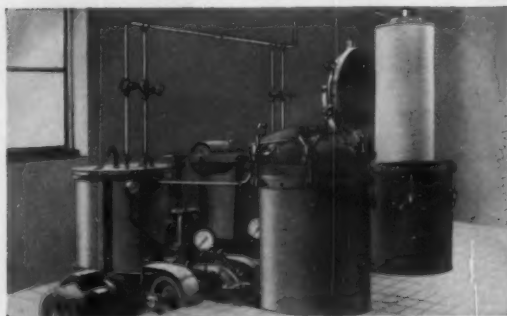
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FORTHCOMING MEETINGS OF THE SOCIETY—continued from page x

Tuesday, 5th February 1957

MANCHESTER JUNIOR BRANCH. "... and inwardly digest". A Dissertation on the difficulty of assimilating Technical literature. S. Burgess, Esq., A.M.C.T., F.S.D.C. (Courtaulds Ltd., Droylsden Dyehouse), Manchester College of Science and Technology, Manchester. 7 p.m.

Tuesday, 12th February 1957

SCOTTISH SECTION. *Reactive Dyes—A New Approach to Cellulose Dyeing*. R. W. Speke, Esq., B.Sc. (I.C.I. Ltd.). St. Enoch Hotel, Glasgow. 7.15 p.m.

Wednesday, 13th February 1957

NORTHERN IRELAND SECTION. *Fifty Years Dyeing Linen in Ireland*. N. McCraith, Esq., B.Sc. (Joint Meeting with Textile Institute.) Thompson's Restaurant, Donegall Place, Belfast. 7.30 p.m.

BRADFORD JUNIOR BRANCH. *Finishing of Garments and Fabrics made from Bulked "Terylene" Yarns and Filaments*. G. McLewy, Esq., B.Sc., Ph.D., A.R.I.C. (I.C.I. Ltd. Terylene Council). Technical College, Bradford. 7.15 p.m.

Thursday, 14th February 1957

WEST RIDING SECTION. *Applications of Colour Physics to Textiles*. J. C. Guthrie, Esq., M.Sc., F.Inst.P., F.T.I. P. H. Oliver, Esq., B.Sc., Grad.Inst.P. (Courtaulds Ltd.). Hotel Metropole, King Street, Leeds. 7.30 p.m.

Friday, 15th February 1957

MANCHESTER SECTION. *Problems in the Wet Processing of Rayon Blends*. H. D. Edwards, Esq., Ph.D., A.R.I.C. and R. C. Cheetham, Esq., A.M.C.T., F.R.I.C. (Courtaulds Ltd.). Textile Institute, 10 Blackfriars Street, Manchester. 7 p.m.

Tuesday, 19th February 1957

HUDDERSFIELD SECTION. *Petroleum Chemicals in the Textile Industry*. H. Bratherton, Esq., A.M.C.T., A.T.I. Silvios Cafe, Westgate, Huddersfield. 7.30 p.m.

Wednesday, 20th February 1957

MIDLANDS SECTION. *Basic Mechanisms of Winch Processing*. R. H. Waddington, Esq., B.Sc. Albert Hall Institute, Nottingham. 7 p.m.

Thursday, 28th February 1957

WEST RIDING SECTION. *The Applications of Fluid Beds to Dyeing*. J. Wilson, Esq., M.C., M.Sc. and F. G. Audas, Esq. (British Rayon Research Association). Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Tuesday, 5th March 1957

LEEDS JUNIOR BRANCH. *Reactive Dyes. A New Approach to Cellulose Dyeing*. C. D. Weston, Esq. (Imperial Chemical Industries Ltd.). Departmental Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, Leeds 2. 3.30 p.m.

MANCHESTER JUNIOR BRANCH. *The Principles and Practice of Package Dyeing*. K. Limbert, Esq. (The Longclose Engineering Co. Ltd., Leeds). Manchester College of Science and Technology, Manchester. 7 p.m.

Thursday, 7th March 1957

BRADFORD JUNIOR BRANCH. Lecture. Details later.

Friday, 8th March 1957

LONDON SECTION. *Soiling of Natural and Synthetic Fibres*. G. G. Taylor, Esq., B.Sc., A.Inst.P. (Clayton Dyestuffs Co. Ltd.). Royal Society, Burlington House, London W.1. 6 p.m.

Tuesday, 12th March 1957

SCOTTISH SECTION. *Recent Developments in Wool Dyeing*. G. H. Lister, Esq., Ph.D., B.Sc. (Sandoz Products Ltd.). St. Enoch Hotel, Glasgow. 7.15 p.m.

Wednesday, 13th March 1957

NORTHERN IRELAND SECTION. *Problems in Wet Processing of Rayon Blends*. H. D. Edwards, Esq., Ph.D., A.R.I.C. and R. C. Cheetham, Esq., A.M.C.T., F.R.I.C. (Courtaulds Ltd.). Thompson's Restaurant, Donegall Place, Belfast. 7.30 p.m.

Thursday, 14th March 1957

WEST RIDING SECTION. *The Dyeing of Acrylic Fibres*. K. Meldrum, Esq., B.Sc., A.R.I.C. (Courtaulds Ltd.). Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Tuesday, 19th March 1957

HUDDERSFIELD SECTION. Annual General Meeting. Silvios Cafe, Westgate, Huddersfield. 7.30 p.m.

Wednesday, 20th March 1957

MIDLANDS SECTION. *Developments in Sizing as they affect the Dyer and Finisher*. J. H. MacGregor, Esq., Ph.D., F.R.I.C., and E. France, Esq., A.R.C.S., A.M.C.T. (Joint with the British Association of Chemists.) Midland Hotel, Derby. 7 p.m.

Friday, 22nd March 1957

BRADFORD JUNIOR BRANCH. Annual Dance. Connaught Rooms, Bradford.

Saturday, 23rd March 1957

NORTHERN IRELAND SECTION. Annual General Meeting and Dinner. Woodbourne House Hotel, Belfast. Details later.

Thursday, 28th March 1957

MIDLANDS SECTION. *The Soiling of Natural and Synthetic Fibres*. G. G. Taylor, Esq., B.Sc., A.Inst.P. (Joint with the Textile Institute.) Carpet Trades Canteen, Kidderminster. 7.30 p.m.

WEST RIDING SECTION. Annual General Meeting. Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Friday, 29th March 1957

The Society's Annual Dinner. The Grosvenor House, Park Lane, London.

* Tuesday, 9th April 1957

SCOTTISH SECTION. Annual General Meeting. 7 p.m. Followed by ordinary Meeting at 7.30 p.m. *Developments in Sizing as they Affect the Dyer and Finisher*. J. H. MacGregor, Esq., Ph.D., F.R.I.C. and E. France, Esq., A.R.C.S., A.M.C.T. (Courtaulds Ltd.). St. Enoch Hotel, Glasgow.

Friday, 12th April 1957

LONDON SECTION. *Some Aspects of Bleaching with Hydrogen Peroxide and Peracetic Acid*. L. Cheeser, Esq., B.Sc., A.T.I. and G. C. Woodford, Esq. (Laporte Chemicals Ltd.). George Hotel, Luton. 7 p.m.

MIDLANDS SECTION. Annual Dinner. George Hotel, Nottingham. 7 p.m.

Wednesday, 17th April 1957

MIDLANDS SECTION. "... and inwardly digest (a Dissertation on Technical Literature). S. Burgess, Esq., F.S.D.C., A.M.C.T. (Preceded at 6.30 p.m. by the Annual General Meeting of the Section). King's Head Hotel, Loughborough. 7 p.m.

Saturday, 20th April 1957

BRADFORD JUNIOR BRANCH. Annual General Meeting. Technical College, Bradford. 10.15 a.m.

Friday, 3rd May 1957

MIDLANDS SECTION. The Mercer Lecture of the Society. Grand Hotel, Leicester. 7 p.m.

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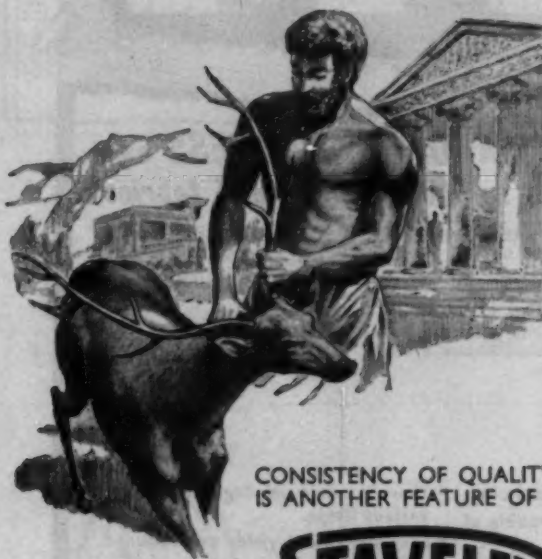
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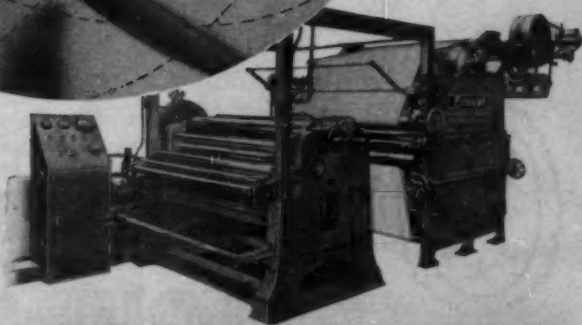
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